Chemical & Process Engineering

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TOPICS OF THE MONTH 37

CORROSION FOR CHEMICAL ENGINEERS—I. Electrochemical Principles *by L. L. Shreir, Ph.D., F.R.I.C., F.I.M.* 41

BIOCHEMICAL ENGINEERING *by J. J. H. Hastings, M.B.E., M.Sc.(Tech.), A.R.I.C.* 46

CORRESPONDENCE 47

LARGE-SCALE PRODUCTION OF LIQUID GASES ... 48

RECENT PUBLICATIONS 50

CHEMICAL and ENGINEERING RESEARCH in AUSTRALIA 51

CARBON DIOXIDE BY MOLASSES FERMENTATION ... 55

ENRICHMENT OF INDUSTRIAL GASES BY METHANE SYNTHESIS 56

CANADIAN FERTILISER MANUFACTURERS BUILD ANOTHER PLANT 57

EFFLUENT TREATMENT AT A FINE CHEMICAL FACTORY 59

PLANT AND EQUIPMENT:
Measuring oxygen and hydrogen in gases; Valve position indicators; Flexible metallic couplings; Lighting system for hazardous atmospheres; Small continuous fat-splitting plant; New exhaust equipment 61

CHEMICAL ENGINEERING INVENTION 63

VINYL RESIN MANUFACTURE 64

EXPORT OPPORTUNITIES 65

PERSONAL PARAGRAPHS 66

NEW STANDARDS 66

WORLD NEWS:
From Great Britain, Germany, Austria, Mexico, Spain, Southern Rhodesia, Australia, Sweden, France, Canada, India, Iraq, Venezuela, Netherlands, U.S.A. 67

MEETINGS 70

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Topics of the Month

Chinese chemical research

THE difficulty of getting accurate information on industrial developments in Communist countries is well known. Such information is almost invariably mixed up with political propaganda and any indications of output are mostly given as meaningless percentages. However, it is important to gather and assess whatever crumbs of information come our way and for this reason we were interested in a recent article on the Chinese chemical industry and its research interests in a Russian periodical. No production figures are given, but there is a survey of research projects which indicates the present pattern of Chinese chemical effort. Apart from the analytical and product research laboratories of individual factories, there are now 11 chemical research establishments in China—hardly an impressive number in comparison with almost any Western country. Five of these establishments are associated with the Academy of Science, and they vary appreciably in size with staffs ranging from about 50 to several hundred.

Research is concerned primarily with liquid fuels, dyes, rubber, synthetic resins and plastics, petroleum chemicals, lubricants, abrasives, ceramics, catalysis, and pharmaceuticals including antibiotics. In the field of petroleum research, attention is focussed on catalytic cracking and the aromatisation and complete polymerisation of cracking gases. Lubricant additives are also being studied. Research on shale oils and coal tars is also designed to produce fuels and lubricants.

Attempts are being made to build a synthetic rubber industry and the production of carbon blacks from indigenous fuels is being considered. In the allied field of plastics, phenol formaldehyde, polyvinyl and organo silicon compounds are being investigated. Products range from acid-resistant linings to insulating varnishes.

The inorganic chemical industry is interested in the recovery of iodine and potassium from sea water, soda manufacture by the Ko-Deban method, the production of potash salts and borax in north-west China, the study of alkaline metal sulphides and the salts of cobalt, vanadium and thorium. Attempts are being made to improve vanadium catalysts for sulphuric acid manufacture.

Cellulose, rare elements, refractories, pesticides and disinfectants are other products on which the Chinese are conducting research.

Slurry pelleting process

A PROCESS for pelleting colliery slurry so that it can be used as a fuel or for making producer gas, has been developed by the British Coal Utilisation Research Association. The plant needed consists essentially of a disintegrator and a grooved, oscillating pelleting tray. A rating of up to 1 ton/hr. has been obtained on a tray 5 ft. long by 1 ft. wide. Pellets of size ranges $\frac{1}{8}$ to $\frac{1}{4}$ in. or $\frac{1}{4}$ to $\frac{1}{2}$ in., strong enough to withstand moderate handling, are produced. A number of slurries of varying coal and ash characteristics have been successfully pelleted.

Further work is in hand on the pre-treatment of the slurry feed and on improving the method of drying the pellets. Extension of the pelleting process to materials other than colliery slurry is also under investigation. There are many industrial substances, such as sludges and 'sticky' wastes, which have pelleting possibilities.

Boiler-firing is an attractive use for the slurry pellets which has been tried out on an Economic boiler fitted with a chain-grate stoker. It was found that pellets can be successfully used for steam-raising with an efficiency and rating higher than is possible with raw slurry. The boiler used by the B.C.U.R.A. has a rating of 1 ton/hr.; the National Coal Board are also experimenting with slurry pellets for boiler-firing.

Another possibility is the use of slurry pellets for making producer gas for firing coke ovens. Trials were made on a semi-technical gasification unit. It has been revealed that pellets can be successfully gasified in a conventional fuel bed, but a high steam/air ratio in the blast is necessary to prevent clinkering. A further investigation, carried out at Sheffield, was concerned with the gasification of slurry. It was found that the pressure drop in a bed of slurry can be reduced by a third by removing 8% of the material from the base of the bed.

Acoustic gas indicator

A GAS indicator which will operate in any mixture of gases having different sound velocities is expected to find many uses in the gas and chemical industries. Known as the acoustic phase shift gas indicator, it has been designed by Drs. L. E. Lawley and E. G. Richardson of King's College, Newcastle, for use at a colliery where methane is extracted from boreholes near the workings and piped to the surface.

The methane is used to save coal by burning it under the colliery boilers, but if air were to leak into the system in certain proportions an explosion might occur. The apparatus indicates the proportion of air in the mixture and gives an immediate warning when the air content exceeds a certain amount.

The advantages of this instrument over those now used are that it operates instantaneously and can be used safely even in an explosive gas mixture. It works like this:

A mixture of two gases is passed through a short tube which contains a telephone earpiece emitting a continuous note at one end and a microphone at the other. When the proportions of the gas mixture change the speed of a sound wave through the gas changes. The cathode ray indicator shows the relative time difference between a particular part of the sound wave leaving the telephone earpiece and arriving at the microphone. This time difference varies with the speed of sound through the gas and hence with the proportions of the gas mixture.

When the proportion of one of the gases exceeds a certain amount a loud buzzer sounds, a light glows on the indicator unit and external contacts are provided so that the gas supply can be automatically switched off. The warning operates instantaneously and remains on until the proportions of the gas mixture are corrected. It can then be reset by hand.

The indicator may be switched off, leaving the warning system still operative. In this case the indicator switches itself on automatically when the warning operates. A switch is also provided to enable the warning system to be completely checked whatever gas mixture is in the tube.

Ten thousand corrosion tests

OVER 200,000 catalogued items of information about corrosion have been accumulated as a result of more than 10,000 tests carried out by International Nickel Co. of Canada Ltd. This information is made available by the company by direct consultation, in publications, and through lectures by members of its staff. These are not confined to the properties of nickel alloys, but extend to discussions of the basic principles of the causes and prevention of corrosion. A colour film with sound, 'Corrosion in Action,' which illustrates how corrosion occurs and how it can be controlled, is being shown to technical, industrial and educational groups.

It is almost half a century since Inco first undertook to determine the corrosion-resisting properties of pure nickel and nickel-containing alloys. Thirty-three years ago the company established a Fellowship for the study of corrosion at the Mellon Institute of Industrial Research at Pittsburg, Pennsylvania, and an early achievement was the solution of certain difficult corrosion problems in steel mill pickling operations through the recognition of 'concentration cells' and their effect. Since the Company's Bayonne, New Jersey, research laboratory was established, one of its principal activities has been the study of corrosion. In the U.K., the Mond research laboratories in Birmingham and Acton have given similar attention to the subject.

Some years ago laboratory studies were extended to corrosion investigations under natural conditions at several field testing stations. These provide for exposure of specimens to widely different atmospheric effects at Basking Ridge, Jersey City and Bayonne, New Jersey, and at Block Island, Rhode Island. Research on the behavour of materials in salt water and sea air is conducted at Kure Beach and Harbour Island on the North Carolina coast near Wilmington. The facilities at North Carolina have been used by more than 100 producers of sometimes competitive materials, government agencies, educational institutions, and by engineers and scientists from many parts of the world. The number of specimens now exposed in sea water is more than 2,500; the total for the past 18 years is over 10,000. At present there are about 25,000 specimens undergoing atmospheric tests.

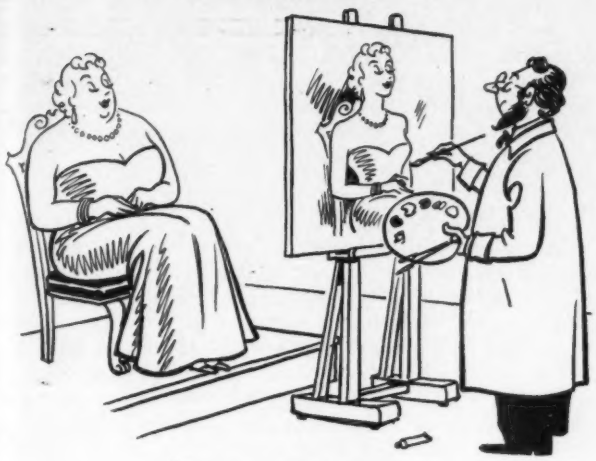
Automatic control of nuclear reactors

WHAT is claimed to be the first complete automatic control system for nuclear reactors and their ancillary plants has been designed in the U.S. Designed by the Minneapolis-Honeywell Regulator Co., it will include servo-amplifiers, recorders, controllers and servo-motors for the automatic regulation of the rate of power generation of nuclear reactors.

The initial function of the control system is to ensure a safe start-up of the chain reaction. This commences when the nucleus of a uranium atom is split under neutron bombardment. As it splits it releases more neutrons. These, in turn, split additional nuclei, releasing more neutrons, thus producing the reaction. To sustain this reaction, x number of neutrons must be liberated per second.

As the control rods are removed to begin the chain reaction, a neutron-sensitive detector in the reactor itself emits electronic pulses. These are picked up by an electronic amplifier—one pulse for each neutron detected by the chamber. Another electronic device counts these pulses, totalling them on a digital register. The operator, by deter-

Comical Engineering Terms



"SIZE REDUCTION"

mining the number of pulses occurring per second (or any given period), knows the rate at which the reaction is increasing and adjusts the control rods accordingly. As the reaction increases, still another electronic instrument, a counting rate meter, integrates these pulses, translating them into voltage readings proportional to the number of pulses detected per second. The overall effect is to provide the operator with an automatic means of sensitively regulating the early stages of the chain reaction.

During this entire start-up process, and after the proper operating level has been reached, other electronic units in the control system are continually monitoring the reaction. For example, one series of electronic amplifiers and recording instruments monitors the reaction for too rapid a rise in power level. If the units detect any abnormality they immediately shut down the reactor. Other amplifiers prevent the power level from going too high. Should this occur they also will immediately shut down the reactor.

Once the reactor is safely started and at the desired operating level, a final control series takes over to regulate continuously the chain reaction at predetermined levels. To accomplish this, a special instrument called an electrometer records the power level, signalling servo-amplifier units. These units, linked to special servo-motor equipment, will automatically adjust the control rods to regulate the nuclear reaction.

Polythene in the tropics

THE first reliable results on the behaviour of polythene under tropical conditions have been obtained in tests carried out by the Ministry of Supply and the British Plastics Federation. These tests are particularly important, since polythene is used for a variety of high-frequency electrical applications because of its low power factor and permittivity. Because of its ductility and inertness it is also suitable for such purposes as packaging and water piping.

Two grades of polythene, each sample of which contained antioxidant (0.1% of *Nonoxol* D.C.P.) but which were present both with and without the inclusion of carbon black (0.1% *Kosmos* B), were exposed for periods of three, six or twelve months to four sets of conditions—jungle undergrowth, jungle clearing, semi-desert and tropical surf beach—in Nigeria, whilst those specimens requiring special test equipment were returned to London in hermetically-sealed containers.

It was found that polythene is resistant to the moisture of tropical climate as represented mainly by the jungle exposure sites, but is susceptible to the actinic effects of the tropical sun, preponderant on the beach and desert exposure sites. These effects, however, are greatly reduced by carbon black (which is specifically recommended for this purpose by the makers of polythene).

The main results of the continued sun exposure were: an increase in 'brittleness,' exemplified by loss of flexibility and extensibility, a rise in cold bend temperature and the appearance of surface cracks; and a thirty-fold rise of power factor in the absence of carbon black or ten-fold rise in the presence of carbon black, under the worst conditions of the trials described.

As ductility and low power factor were the foremost properties leading to the selection of polythene in preference to any other material, it is concluded that it would be necessary either to be satisfied with a limited service life in direct sunlight, or to provide additional protection for the material. Full details of the test are given in the report, 'Reports on Plastics in the Tropics—2: Polythene,' H.M.S.O., 2s. net.

Gas from fuel oil

A NEW plant for producing gas from residual fuel oil too heavy for general use by transport has been developed by the South Eastern Gas Board. The gas is very similar in composition to ordinary town gas, and the first full-scale commercial plant, which has been installed at Lower Sydenham, London, is producing up to 800,000 cu. ft. of gas and saving 60 tons of coal a day.

The development was initiated by the former South Metropolitan Gas Co. It was found that certain catalysts could so guide chemical reaction between steam and heavy oil that a gas was produced similar in composition to town gas. It was later shown that the same catalytic materials could be used to adjust the quality of both refinery tail gases and methane.

The prototype plant takes in 130 gal./hr. of oil and produces 1.15 therms of gas a gallon. Although the oil is a little more expensive than coal, the cost of producing a therm compares closely with that of the orthodox method.

Salt mining and processing in the Netherlands

THE discovery of new and extensive salt deposits in the Winschoten district of Holland, estimated at around 20,000 million tons, serves as a reminder of the somewhat unusual method of winning salt which has had to be adopted in the Netherlands because of great depth at which the seams lie. Some of these are 300 to 400 metres below ground level and the normal method of underground cutting of rock salt would be far too costly.

Although still expensive enough the Dutch method of injecting water into the seams and forcing the resulting brine to the surface has the advantage that refined salt is produced in one operation by the evaporation of the saline solution.

Found more or less accidentally in both the regions that have produced salt in Holland, namely at Boekelo, in the province of Overijsel, and Hengelo, the rock salt deposits have been discovered at depths of from 325 to 400 metres. Centuries ago they were formed from salty inland seas or

lakes, which, having reached saturation point through evaporation, caused the concentration, crystallisation and deposition of the salt on the floor of the sea. In course of time these beds reached considerable proportions, became part of the earth's crust and, under enormous pressure, lost their moisture content and became rock salt.

Percussion drilling and rotary drilling with a diamond crown have both been applied to the borings of the K.N.Z. (Koninklijke Nederlandsche Zout Industrie or Royal Netherlands Salt Industry). As soon as the drill reaches the bed of salt a steel tube is extended downwards just as far as the upper side of the bed and hermetically sealed in the rock. A thinner tube is then introduced into this and taken down to the bottom extremity of the salt. Water, withdrawn by artesian well from a water bearing strata, is now pumped down the larger pipe, makes a saline solution with the rock salt and is forced to the surface up the inner narrower tube. The saline solution is then carried under pressure to the factory for refining. Excise duty has to be paid on the salt recovered and this is based on the salt content of the brine as it stands in gauged reservoirs.

As produced by K.N.Z. the refined salt has a regular crystal pattern, produces a clear solution when dissolved and contains 99.8% NaCl. In this it differs from crushed rock salt which has no regular crystal pattern and contains 1 to 2% of insoluble matter.

From an output of 25,000 tons in 1920 salt production reached a figure of 481,125 metric tons in 1951; comparing with 412,092 metric tons in 1950. In 1951 some 27,000 tons were processed into chemical products. The rise in production was due to a great extent to more efficient use of the capacity of existing installations.

The salt deposits at Winschoten are claimed to have a thickness of 1,000 metres and to extend over a width of several kilometres. They are expected to be worked similarly to the existing fields and the salt recovered will be used entirely for the manufacturing of soda and soda products. A soda factory is to be set up at Delfzijl, a North Sea port. Kept in its fluid state the brine solution will be fed to the factory by pipeline from the borehole. The capacity of the factory will be about 500 tons/day of soda and it will employ 400-500 workers. American technicians will co-operate in designing, constructing and starting up the factory. The cost of the whole project including harbour installations and the laying of pipelines, will be about £5 million.

Smelling danger

THE contribution to coal mines safety represented by the acoustic gas indicator, described in another column, reminds us of another scientific colliery safeguard, this time devised by chemists. The idea is simple; it is to create a strong, pungent smell when colliery machinery gets overheated and thus liable to catch fire. This is done by embodying a stench agent in a special capsule which is attached to the machine. The capsule is sealed with a plug formulated so that it melts at a chosen temperature and releases the stench which travels through the ventilation of the mine and thus signals danger.

The stench fire warning is used in mines in Ontario but in Britain the idea has been taken up hitherto only by British Railways who use it to warn engine drivers of overheated bearings on their locomotives. Now the Safety in Mines Research Board have adopted it for use in pits. Their chemists are trying to find the most penetrating and far-spreading stench agent—an unusual job for a chemist who in this connection is generally asked to neutralise smells or find perfumes. The champion smell-raiser so far is butyl mercaptan (skunk and garlic odour) which has produced a strong smell up to at least 1.7 miles from the discharge point. *Beta*-ionone (violets) could be smelt only a mile away in this trial. Further experiments are under way to find better (or worse) smells.

Gypsum may solve India's sulphur problem

THE recent world shortage of sulphur has brought home to India's sulphur-consuming industries, as to those of other countries, the drawbacks of being dependent on foreign—mainly American—sources. No domestic sulphur is produced in India, nor are there any brimstone deposits. By 1955-56, at the end of the first five-year plan, India's present requirements of about 65,000 tons p.a. are expected to rise to 85,000 tons and thereafter, say a decade hence, can be conservatively put at 100,000 tons. For the developing Indian chemical industry, is seems essential that dependence on imports be reduced.

With these considerations in mind, H. N. Ganguli, in a recent issue of the Indian *Chemical Age*, discusses possible alternative sources of sulphur in India. He predicts production targets, within the next decade, of 30,000 to 40,000 tons p.a. of sulphuric acid from pyrites, and 10,000 to 15,000 tons p.a. of sulphur recovered from waste industrial gases and effluents. This would still leave a gap of between 35,000 and 50,000 tons of sulphur to be obtained from other sources in order to achieve self-sufficiency.

It is here that the utilisation of *gypsum* as a raw material for sulphur production assumes great significance for India. Sulphur (or sulphuric acid) can be obtained from gypsum or anhydrite by the Mueller-Kuehne process, which has become well known in Europe since the first world war. Apart from the original plant of Farbenfabriken Bayer A.G. at Leverkusen, Germany, large units suitable for this process have been set up at the former I.G. works at Wolfen, Germany, at I.C.I.'s works at Billingham, England, and in the state-owned works at St. Chamas in France. Further plants are under construction in various countries. The Russians, as pointed out in last month's CHEMICAL & PROCESS ENGINEERING, are also interested in a similar process.

In addition to gypsum or anhydrite, the process utilises clay, iron ore and pyrites ash as other raw materials, and solid, liquid or gaseous fuel. What gives the process such distinct possibilities in India is that all the raw materials are available in suitable locations and in convenient form. Another attractive feature of the process is that it yields not only sulphur, but also cement as a by-product, the source of calcium being gypsum instead of the conventional limestone. An annual production of 30,000 tons of sulphur utilising about 200,000 tons of gypsum, will give as by-product about 400 tons/day of cement.

The gypsum-sulphur process can produce elemental sulphur economically, although no doubt the end-product is obtained more economically as acid than in elemental form. In some parts of India, the regional demands for both sulphuric acid and cement might make the production of acid from gypsum worth while. In most areas, however, owing to the structure of the sulphuric acid and other sulphur-consuming industries, and the difficulty of transporting acid, the fairly large-scale production of elemental sulphur from gypsum would prove more economic.

CORROSION FOR CHEMICAL ENGINEERS

I. Electrochemical Principles of Corrosion

By L. L. Shreir, Ph.D., F.R.I.C., F.I.M.
(*Battersea Polytechnic, London*)

The inherent tendency of the common metals to corrode in various environments is a problem which continually faces the chemical engineer. More awareness of the importance of corrosion and the increased use of scientific principles in its control could save enormous sums of money by conserving metals and reducing expenditure caused by plant failures. With this in mind, we have commissioned this series of articles in which the author will discuss concisely the basic principles of corrosion with particular emphasis on chemical engineering aspects. This first article is confined to electrochemical considerations, as these form a theoretical background for the subsequent articles which will be of a more practical nature.

CORROSION may be defined as the destruction of metals by chemical and electrochemical agencies, and is essentially the reverse process to that involved in reducing metals from their ores. A consideration of the standard free energies of formation of metal oxides[1] shows that, with the exception of certain noble metals, this is a spontaneous process, being accompanied by a decrease in free energy. Hence corrosion is the thermodynamically probable process, and the corrosion product rather than the metal is the stable form. However, many of these reactive metals are relatively stable in practice, so that it is evident that, although thermodynamics gives a criterion of the tendency of a corrosion reaction to proceed, the actual process is frequently determined by other considerations.

Corrosion is always the result of the interaction of the metal and its environment and, hence, in considering the mechanism of the process, and methods of protection, both must be taken into consideration. The result of corrosion is the formation of a chemical compound, and it is the nature, physical condition and position of formation of this compound which determine the subsequent course of the reaction.

Considering the large variety of metals and alloys available, and the infinite number of possible corrosive environments, it is evident that the subject is very extensive. However, there are certain basic electrochemical principles applicable to most corrosion reactions, an understanding of which is essential in deciding upon the practical methods for preventing corrosion.

Types of corrosion

For convenience, corrosion processes may be divided into two classes, depending on the presence or absence of an electrolyte:

(i) Dry Corrosion.

(ii) Electrochemical Corrosion.

The direct combination of metals with oxygen, sulphur and chlorine, etc., are examples of the former process, but it should be realised that in many cases these reactions occur also by a galvanic cell mechanism and are hence electrochemical.

The corrosion of metals in the atmosphere, immersed in natural waters, or chemical solutions, or buried underground, are examples of electrochemical corrosion and involve the presence of an aqueous electrolyte.

Dry corrosion

The formation of oxide films when metals are heated in air is the most important type of dry corrosion. Even at room temperature the common metals form appreciable oxide films which, however, are transparent.

At higher temperatures these transparent films thicken and are responsible for the interference tints or temper colours produced when iron is heated. Isolation of these films on metals by U. R. Evans and others has been a notable contribution to the study and understanding of corrosion phenomena.[2]

This chemical reaction is typified by the fact that the oxidation-reduction process and the formation of the oxide film occur in one and the same place. The oxide film thus forms a barrier which, depending on its physical nature, may stifle further reaction or allow it to proceed. Metals like sodium, potassium and calcium form porous films which are thus non-protective, whilst the continuous adherent thin films formed on aluminium, stainless steel, titanium, nickel, monel, tin, chromium, lead, zinc and iron are protective.[3]

A quantitative study of film growth shows that for many metals the oxidation-time curves follow a parabolic law, the rate of oxidation decreasing with time. These protective films have been found to be electrical semi-conductors and to allow the passage of both electrons and ions, thus acting both as electronic and electrolytic conductors.[4] Film growth develops at the oxygen/oxide film interface,[5] and is due to the migration of metal ions and electrons to this surface and the combination of the metallic ion with the oxygen ionised by the electrons.

These oxides appear to have more or less metal than corresponds with the stoichiometric formula.[6] Thus FeO, NiO and Cu_2O are examples of metal-deficit oxides which have vacant metal sites on the oxide crystal lattice which permit ionic migration; ZnO, BeO, MgO and Fe_2O_3 are examples of metal excess oxides in which the excess metal ions occupy interstitial lattice positions.

Thus, according to modern conceptions of film growth, the process in many cases is essentially electrochemical and occurs by a galvanic cell mechanism.[7]

It is apparent, therefore, that the sharp distinction between dry oxidation and electrochemical corrosion no longer holds, as both are characterised by an electrolyte.[8] However, it must be emphasised that this is not always so and, in many cases, dry oxidation occurs by inward diffusion of oxygen.[9]

Electrochemical corrosion

This is characterised by the presence of an aqueous electrolyte. It is a generally accepted fact that it occurs by a galvanic cell mechanism where oxidation occurs at one electrode and reduction at the other, whilst the corrosion product, if insoluble, may form at some intermediate position. In this case it will not stifle the attack as

in the case of dry corrosion; it is obvious that this type or corrosion can be particularly dangerous.

The essential feature of electrochemical corrosion can be illustrated by reference to the Daniell cell (Fig. 1) in which the spontaneous reaction:

$$Cu^{++} + Zn = Cu + Zn^{++} \quad \ldots\ldots (1)$$

proceeds with the production of electrical energy. Oxidation of the zinc proceeds at one electrode and reduction of cupric ions to copper occurs at the other; it is a convention in corrosion cells to regard the former electrode as the *anode* and the latter as the *cathode*. It follows that electrochemical corrosion is a process of oxidation occurring in a galvanic cell at the anode, whilst reduction and hence protection occurs at the cathode.

In addition to the electrode reactions, there will be migration and diffusion of the ions through the electrolyte and migration of electrons through the metallic conductor. All these processes must operate simultaneously for corrosion to occur, and any factor which reduces the velocity of one will affect the corrosion rate, which is determined by the magnitude of the current, and follows Faraday's laws. Thus the formation of a corrosion product, adjacent to either the anode or cathode, will tend to reduce the corrosion rate.

Thermodynamics of immersed corrosion

The free energy change gives the tendency of the reaction to go to equilibrium and, although it takes no account of kinetics, it is of value as giving a useful measure of the initial susceptibility of a corrosion process.

For a reversible cell under equilibrium conditions, $-\triangle G = nFE_r$, where n is the number of gramme equivalents involved in the process, F is the Faraday (96,500 coulombs) and E_r is the reversible e.m.f. of the cell. The latter can be evaluated from the standard electrode potentials* (Table 1), in which the potential of a metal in a solution of its ions at unit activity is determined relative to the standard hydrogen electrode, which is given an arbitrary zero potential:

$$2H^+ + 2e = H_2$$
$$a = 1 \quad p = 1 \text{ atm.} \quad E^\circ = 0.00 \text{ volts.}$$

*The sign of the potential is arbitrary and, in Table 1, the Continental convention is adopted where the potential of the metal is taken relative to the electrolyte and hence can be regarded as a reduction series:

$$Cu^{++} + 2e = Cu$$
$$E^\circ = +0.34 \text{ volt.}$$

(The convention usually adopted in the U.S.A. takes the potential of the solution relative to the metal and can be regarded as an oxidation series:

$$Cu = Cu^{++} + 2e$$
$$E^\circ = -0.34 \text{ volt.})$$

To avoid confusion the terms 'noble' (cathodic) and 'base' (anodic) will be adopted for the electropositive and electronegative metals, respectively.

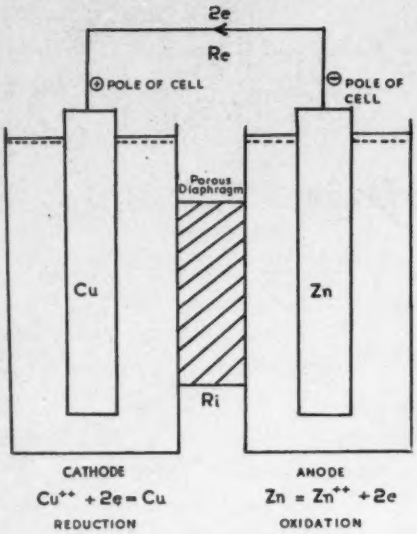


Fig. 1. The Daniell cell.

For the Daniell cell, the reversible e.m.f. will be the algebraic sum of the equilibrium potentials of the two half cells and, assuming the ions are at unit activity:

$$E_r = 0.34 - (-0.76) = 1.10 \text{ volts}$$
$$\therefore -\triangle G$$
$$= 2 \times 96{,}500 \times 1.10 \text{ volt coulombs/mol.}$$
$$= 212{,}300/4.1840 = 50{,}740 \text{ cal./mol.}$$

If the equilibrium potential is known, the free energy can be obtained, or conversely.

In a similar way the corrosion of metals by acid can be regarded as a process occurring in a galvanic cell where $2H^+ + 2e = H_2$ is the cathodic reaction and $M = M^{++} + 2e$ is the anodic reaction. It is apparent that for metals below hydrogen (*i.e.* more anodic) $-\triangle G$ for the reaction $M + 2H^+ = H_2 + M^{++}$ is positive, and the reaction is thus spontaneous, whilst the more cathodic metals like Cu and Ag should be unattacked under these conditions.

Table 1. Standard Electrode Potentials.[10]

Electrode reaction	*E° volts*
$Au^{+++} + 3e = Au$	1.42
$Ag^+ + e = Ag$	0.799
$Cu^{++} + 2e = Cu$	0.344
$2H^+ + 2e = H_2$	0.000
$Ni^{++} + 2e = Ni$	−0.23
$Fe^{++} + 2e = Fe$	−0.440
$Zn^{++} + 2e = Zn$	−0.763
$Al^{+++} + 3e = Al$	−1.66
$Mg^{++} + 2e = Mg$	−2.38
$Na^+ + e = Na$	−2.713
$Ca^{++} + 2e = Ca$	−2.84

The potential of the hydrogen electrode is given by:

$$E = E^\circ + \frac{RT}{nF} \log_e a$$
$$= E^\circ + \frac{0.059}{n} \log_{10} a \text{ volts at } 25^\circ C.$$

Thus, in water, pH = 7, $a_{H^+} = 10^{-7}$ and $E = -0.41$ volt, and it follows that the thermodynamic tendency for metals to corrode in pure water with the evolution of hydrogen is considerably reduced. In the case of iron, it is still just positive, but in the case of tin ($E^\circ = -0.13$) and lead ($E^\circ = -0.12$), $-\triangle G$ becomes negative and the reaction is no longer spontaneous.

The electrolysis of water leads to the evolution of hydrogen at the cathode and oxygen at the anode and, although there is no practical reversible oxygen electrode, a theoretical standard electrode potential can be calculated from the energy necessary to decompose water into its elements.

For the reversible oxygen electrode on the hydrogen scale:

$$E = 1.228 - 0.059pH + 0.0147 \log_{10} p_{O_2} \text{ volt at } 25^\circ C. \quad \ldots.(2)$$

Hence, for the theoretical electrode in a solution where $a_{H^+} = 1$ and $p_{O_2} = 1$ atm., $E = E^\circ = 1.228$ volt. In water at pH = 7, it can be seen that $E = 0.815$ volt and, if this is in equilibrium with the air ($p_{O_2} = 0.21$ atm.), this value will only be slightly reduced.

The importance of this cannot be overemphasised, as it shows that the reduction of oxygen occurs at a much more noble potential than the reduction of hydrogen ions to hydrogen gas, and considerably increases the probability of corrosion occurring when dissolved oxygen gas is present. Thus copper which cannot dissolve in hydrochloric acid in the absence of air should do so in the presence of oxygen.

This leads to a classification of corrosion processes according to the cathodic reaction:[11]

(i) Hydrogen evolution type $2H^+ + 2e = H_2$, *e.g.* $Zn(s) + 2H_2O = Zn(OH)_2(s) + H_2(g)$.

Table 2. Thermodynamic Tendency of Corrosion Reactions.[12]

Metal	*Solid corrosion product*	*− ΔG cal./g. mol. of metal*	
		Hydrogen type	*Oxygen type*
Al	$Al(OH)_3$	102,570	180,700
Zn	$Zn(OH)_2$	19,260	75,200
Fe	Fe_3O_4	4,990	79,800
Fe	$Fe(OH)_2$	2,260	58,500
Fe	$Fe(OH)_3$	−6,780	77,800
Ni	$Ni(OH)_2$	−6,760	48,500
Cu	Cu_2O	−10,780	17,400
Cu	$Cu(OH)_2$	−27,900	28,400
Hg	Hg_2O	−21,970	6,190
Ag	Ag_2O	−27,070	1,080

(ii) Oxygen absorption type $\frac{1}{2}O_2 + H_2O + 2e = 2OH^-$, *e.g.* $2Zn(s) + O_2$ (in air) $+ 2H_2O = 2Zn(OH)_2(s)$.

As far as iron is concerned the former reaction will naturally prevail in more acid solutions, whilst the latter will occur in solutions lower in hydrogen ion activity. Under certain circumstances, however, both reactions can occur simultaneously.

Both reactions result in an increase in *p*H, but whereas in acid solutions the hydroxyl ion activity will not exceed the solubility product of the metal hydroxide which thus remains in solution, this will not be the case in neutral solutions. Thus the oxygen absorption type of corrosion is usually accompanied by the formation of insoluble products of the oxides and hydroxides of the corresponding metals. In this case, the thermodynamic calculations will require the standard free energies of these reactions instead of the values previously used for the reaction $M = M^{++} + 2e$.[12] It can be calculated[13] that even metals as noble as Ag, Hg and Cu, although quite resistant to water containing no dissolved oxygen, will tend to corrode spontaneously, forming solid corrosion products in its presence (Table 2).

Irreversible corrosion systems

The previous considerations have been confined to the tendency of reversible cells to go to a state of equilibrium, and it has been shown that the spontaneity of a corrosion reaction can be readily obtained from thermodynamic data. It would appear that in the presence of oxygen most metals will tend to corrode. However, in practice there are many other factors to be taken into consideration and the more important of these are:

(i) Metals are seldom in contact with solutions of their own ions and hence the cells are not usually reversible.

(ii) The corrosion cells are not at equilibrium and hence the electrical energy obtainable is not the maximum.

(iii) The corrosion products can have a pronounced influence in the course of the reaction.

The galvanic series

The galvanic series[14] (Table 3) is based on experiences in corrosion testing, both in the laboratory and under actual operating conditions in numerous corrosives, and on practical results obtained with metals and alloys in service.

This series is of more practical value in engineering design than the table of E° value. The further apart are two metals in this series, the greater will be the likelihood of corrosion of the more anodic member. In addition, it illustrates the pronounced effect of the oxide film on the potentials of metals such as stainless steel, nickel, aluminium, etc. Thus stainless steel in its active form in which the protective oxide film is destroyed, differs only slightly in potential and corrodibility from steel or cast iron. However, in the same environment but in the presence of oxygen or an oxidising agent, the nobility of the metal is considerably increased owing to the presence of a continuous oxide film, and the potential of the metal approaches that of silver.

A further example is aluminium which has an $E^\circ = -1.66$ volt, compared with zinc $E^\circ = -0.76$ volt, but is seen to be more noble than the latter in the galvanic series, owing to the formation of a film of aluminium oxide. This is important in the cathodic protection of steel with coatings of aluminium or zinc.[15]

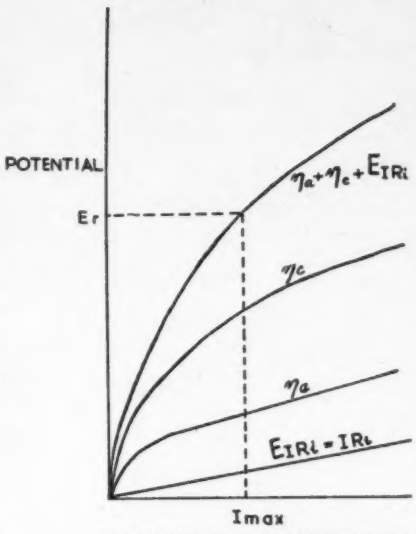


Fig. 2. Factors determining maximum corrosion rate.

Table 3
Galvanic Series of Metals and Alloys[14]

Corroded end (anodic or least noble)

Magnesium
Magnesium alloys
Zinc
Aluminium
Aluminium alloys
Cadmium
Steel
Cast iron
Stainless steel (active)
Lead-tin solder
Lead
Tin
Nickel (active)
Inconel (active)
Brasses
Copper
Bronzes
Copper-nickel alloys
Titanium
Monel
Silver solder
Nickel (passive)
Inconel (passive)
Stainless steel (passive)
Silver

Protected end (cathodic or more noble)

Kinetics of corrosion processes

For a reaction proceeding at a measurable rate in a galvanic cell, there will be a dissipation of electrical energy as heat energy, and hence the e.m.f. will be less than the maximum reversible value. From the point of view of corrosion it should be realised that the maximum current, and hence the maximum corrosion rate, will be limited by these dissipative effects.

These can be classified:

(i) *Electrolytic resistance.* This will depend on the nature, concentration and cross-sectional area of the electrolyte, the latter being important when the cells are set up in the minute pores of metallic coatings. This electrolytic resistance can be considered to set up a back e.m.f.: $E_{IRi} = IR_i$, where R_i is the electrolytic resistance and I the current.

(ii) *Electronic resistance.* In corrosion processes the resistance of the metallic conductor can usually be neglected, as the cell is short-circuited by the corroding metal.

(iii) *Polarisation of the electrodes.* The polarisation of the electrodes is the departure of the potentials from their equilibrium values when the cell works spontaneously at a measurable velocity. Since the velocity of the reaction is proportional to the current flowing, increases in the latter will be accompanied by increase in polarisation. Polarisation is due to concentration changes in the electrode diffusion layer (concentration polarisation) and to irreversible effects due to some stage in the reaction proceeding slowly and thus needing an excess of energy to maintain the velocity.

In the case of gases, this is referred to as an 'overvoltage,' whilst for metal deposition the term 'overpotential' is usually applied.[16]

The cathode is always polarised in the negative direction, whilst the anode is polarised in the positive direction. It therefore follows that, if the potential of a homogeneous metal is more noble than its equilibrium potential, providing that it is not protected by an oxide film, it will be corroding and, conversely, if it is more base, it is *cathodically protected*[30] and will not corrode.

The cell, which will be short-circuited by the corroding metal, will produce a current which, however, will be limited by the back e.m.f., due to the polarised electrodes and by the resistance of the electrolyte. The steady state, or maximum corrosion rate, will be attained when the sum of the dissipative effects, expressed as a back e.m.f., equals the reversible or equilibrium values: $E_r = \eta_a + \eta_c + E_{IRi}$, where η_a is the anode polarisation and η_c the cathode polarisation.

Fig. 2 shows how, by the determination of η–I curves, the corrosion current can be obtained graphically, and illustrates how the rate of change of polarisation with current limits the corrosion rate.[17]

Promoting and controlling factors in galvanic corrosion

The above considerations show that, while E_r, which is the algebraic sum of the

separate equilibrium potentials, is the factor which initiates and promotes the corrosion reaction, it is the irreversible effects which control the subsequent course of the corrosion process.[11]

Promoting factors

A promoting factor is one which will result in the formation of a galvanic cell due to a difference in potential, and can be brought about either by the metal or by its environment. Although it is evident that contact between two different metals may give rise to corrosion, it should be emphasised that any slight heterogeneity on a metal surface may produce galvanic cells and thus promote corrosion. A scratch or break in an oxide film[18] may give rise to an intense attack, as although the anodic area of bare metal is small, the relatively large oxide cathode acts as a 'catchment area' for the reduction of oxygen (Fig. 3), so that the latter process occurs at a low current density and only slight polarisation.[19] The primary corrosion product is $Fe(OH)_2$ and, if sufficient oxygen is present in the NaCl solution, this will be immediately oxidised to rust, *i.e.* $2FeO(OH)$ or $Fe_2O_3H_2O$. If the supply of oxygen is limited the product may be green hydrated magnetite or black anhydrous magnetite, Fe_3O_4. As the corrosion product is formed away from the point of attack, the corrosion reaction will not be stifled as in direct oxidation.

The presence of impurities, differential thermal treatment, different phases in an alloy, differential stress or strain may thus promote corrosion by giving rise to galvanic cells.[20] In the dissolution of metals in acids, it is observed that the attack is generally uniform, owing to the fact that the anode and cathode areas are continually changing. This can be observed when zinc is dissolved in hydrochloric acid by the changing positions of evolution of hydrogen.

Even if the metal is quite uniform, differences in the environment may cause galvanic cells. Thus concentration cells can arise, particularly in underground corrosion, where long lengths of pipe pass through soils of different salt concentration or acidity. It should be emphasised that, in irreversible concentration cells of this type, the metal immersed in the electrolyte of higher concentration is the corroded member, while in reversible cells the anode is the electrode in contact with the more dilute solution.[21] Environmental factors such as different concentrations or compositions, differential heating or illumination and differential agitation may also give rise to corrosion.

Of particular importance in this connection is the 'differential aeration principle' of U. R. Evans,[22] which states that a difference in the oxygen concentration at a metal surface will result in the development of cathodic areas where the oxygen concentration is higher, and anodic areas where it is lower. For a reversible oxygen

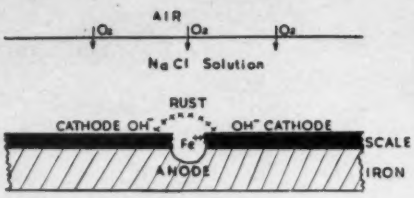


Fig. 3. Corrosion at break in oxide film.

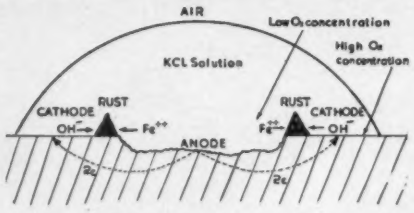


Fig. 4. Differential aeration causing corrosion of iron.

cell, this principle follows from equation 2.

An example of the differential aeration principle is shown in the action of salt droplets on steel, where the oxygen concentration varies owing to the variation of the diffusion of oxygen to the metal through the differing depth of the drop (Fig. 4).

Cathodic controlling factors

Fig. 5 shows that if the cathodic polarisation (η_c) is appreciable, the slope of the cathode potential (E_c) current curve will limit the current, and the process is said to be under cathodic control. This illustrates the danger of the large cathode and small anode (Fig. 3).

In the hydrogen evolution type of corrosion, the cathode process is irreversible, and depends on the nature of the cathode and its true surface area which will control the current density. Table 4[23] shows the values for the hydrogen overvoltage for various metals in 1.0N HCl with a cathode current density of 1 ma./sq.cm., and represents the departure of the electrode from its equilibrium potential in the cathodic direction, although as is customary the negative sign is omitted. Thus the rate of corrosion of zinc in acids is appreciably increased by the presence of impurities of low hydrogen overvoltage,[24] but is decreased by amalgamation owing to high hydrogen overvoltage of mercury. The dissolution of iron is similarly stimulated by contact with platinum.

With the oxygen absorption type of corrosion, insofar as the environment is usually in contact with air and obtains its oxygen supply from this source, the controlling factor will be the rate of diffusion of oxygen to the cathode surface. The depth of immersion of the metal, velocity of flow of the environment, temperature, etc., will all affect the corrosion rate by affecting the diffusion of oxygen.[25] In this connection, the increase in *p*H brought about by this reaction will frequently cause the precipitation of $CaCO_3$ and $MgCO_3$ from hard waters. These porous films will hinder oxygen diffusion to the cathode, whilst the precipitation of salts and rust will stifle the anodic attack.

Resistance control

In Fig. 5 and 6 it is assumed that the conductivity of the electrolyte is sufficiently high for the maximum current to be determined by the intersection of the E_c–I and E_a–I curves. However, if this is not so, and this condition may prevail in natural fresh water, the maximum current is limited by the resistance of the electrolyte R (Fig. 7), *i.e.* $E' = I'_{max}.R$, and I'_{max}. will be less than I_{max}.

Passivity and anodic control

If the polarisation of the cathode is small compared to that of the anode, it is the polarisation of the latter which limits the corrosion, and the process is said to be under anodic control.

Whereas certain metals (*e.g.* Cu, Zn, Ag) dissolve anodically with only a small polarisation, other metals (the transition metals of the periodic table) dissolve with a marked change in potential even at low current densities. This may proceed to such an extent that the more noble potential of the anode permits of an alternative electrode process and results in the liberation of oxygen (or halogen).[26] Under these conditions, the metal, which ceases to be anodically dissolved, behaves like a more noble metal and is said to have become *passive*.

Passivity is thus not an absolute property of the metal and can vary according to the conditions. Uhlig[27] has given two definitions of passivity:

(i) 'A metal active in the e.m.f. series, or an alloy composed of such metals, is considered passive when its electrochemical behaviour becomes that of an appreciably less active metal.'

(ii) 'A metal or alloy is passive if it substantially resists corrosion in an environment where thermodynamically there is a large free energy decrease associated with its passage from the metallic state to appropriate corrosion product.'

Examples of passivity according to definition (i) will include iron in contact with passivators, *e.g.* concentrated nitric acid, potassium dichromate and sodium nitrite. Also included are the transition metals of the periodic classification, and passive alloys like stainless steel which are protected by thin invisible films.

Examples under definition (ii) include those under definition (i) and also passiva-

Table 4. Hydrogen Overpotentials in N-HCl at a Current Density of 1 ma./sq.cm.[23]

Hg	..	1.04 volt	Al	..	0.58 volt	Ni	..	0.33 volt
Cd	..	0.99 „	Cu	..	0.54 „	Pt	..	0.25 „
Pb	..	0.88 „	Ag	..	0.46 „	Au	..	0.17 „
Sn	..	0.85 „	Fe	..	0.40 „	Platinised Pt	..	0.01 „

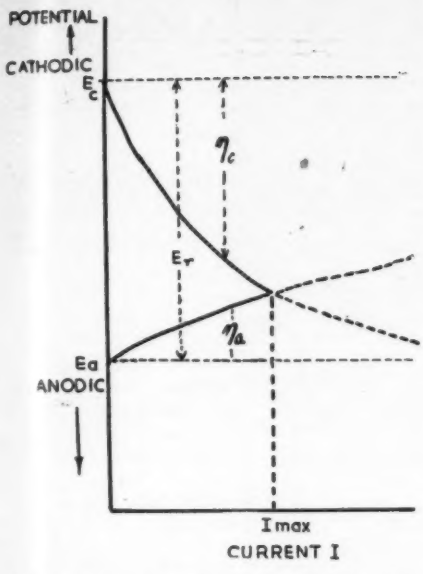


Fig. 5. Corrosion process under cathodic control.

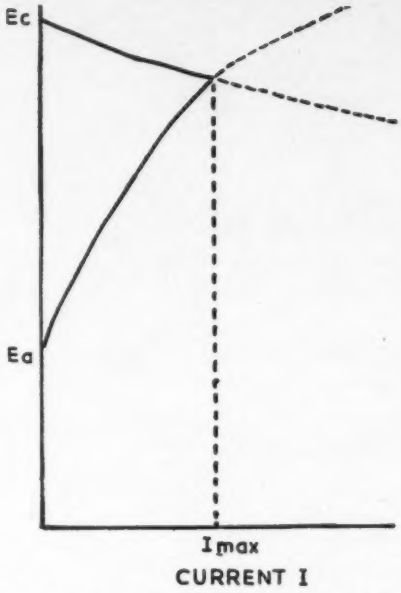


Fig. 6. Corrosion process under anodic control.

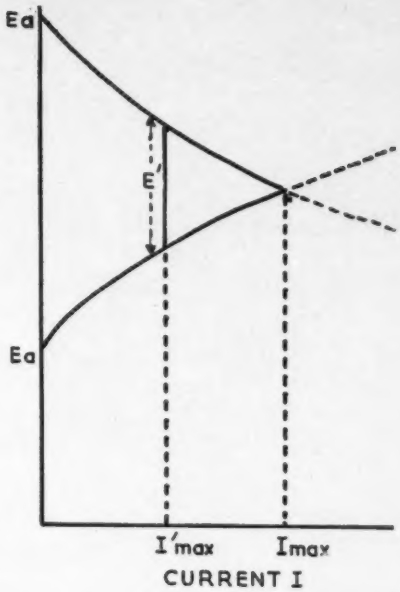


Fig. 7. Corrosion process under mixed control.

tion due to mechanical protection involving relatively thick chemical coatings such as lead in sulphuric acid, silver in hydrochloric acid and iron in concentrated sulphuric acid.

In general, oxidising conditions favour passivity whilst reducing conditions destroy it, causing activity. U. R. Evans has shown that, under certain conditions, iron[28] and zinc[29] can be completely passivated by dissolved oxygen. Halogen ions, particularly chloride, are destructive to passivity, and can be particularly dangerous if the film is destroyed at local points or areas. In this case, the large areas of passive metal combined with the small anodic areas will produce pitting.

It can thus be seen that oxygen plays an important dual role in corrosion processes. Under certain conditions it will stimulate corrosion by providing the necessary cathodic process, whilst under other conditions it will suppress corrosion by producing films at the anode, thus causing passivity.

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Corrosion prevention should begin on the drawing board. In the next article in this series the author will deal with the extremely interesting subject of corrosion and design.

Silicone paints lengthen high-temperature life of metals

Aluminium-pigmented silicone coatings can extend the life of high-temperature equipment and cut fabrication costs. An article in *Materials and Methods*, 1953, **38**, (3), 114, gives these examples:

(1) Silicone-coated mild steel can be substituted for stainless steel in apparatus that operates up to and including 1,000°F. In an oxidation test at 930°F., a panel of untreated mild steel gained in weight by 14 mg./sq.cm. and was still showing an upward trend at the end of the test, whereas a similar panel protected with a silicone-aluminium coating increased by only 2 mg./sq.cm., at which gain it remained constant. Similar tests have been run for 1,000 hr. with no breakdown of the finish.

(2) A silicone-aluminium coating can also lengthen the life of a mild-steel part subjected to intermittent brief exposures to much higher temperatures.

(3) Stainless-steel parts need protection if they are used continuously between 1,400 and 1,600°F. Appreciable protection in this range is provided by silicone-aluminium coatings. In addition to preventing serious oxidation, they promote better heat distribution by reducing local hot spots and in preventing oxidation at welded areas in an assembly.

(4) The life of other heat-resistant alloys can be extended. Tests were carried out on untreated and silicone-coated *Inconel* at 1,400°F. The untreated alloy reached its maximum oxidation—a weight gain of 0.8 mg./sq.cm.—after 8 hr. The silicone-treated alloy reached its maximum oxidation in the same interval, but its average weight gain was only 0.5 mg./sq.cm.

BIOCHEMICAL ENGINEERING

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(The Distillers Co. (Biochemicals) Ltd.)

This is a shortened version of a paper, 'Problems of Biochemical Engineering,' read before the Institution of Chemical Engineers in London recently. The author discusses the engineering problems of the newer fermentation industries with special reference to the manufacture of antibiotics and vitamins. He deals particularly with sterile practices, including the treatment of air, materials, plant and equipment.

'BIOCHEMICAL engineering' means chemical engineering in our newer biochemical industries, *e.g.* the manufacture of antibiotics and certain vitamins, where very large plants are employed to produce micro-quantities of finished products. There were ten principal questions to which a chemical engineer needed answers before a plant for industrial production could be prepared:

(1) *Deep culture.* Would the mould grow and also produce penicillin in a deep layer of aerated liquid?

(2) *Air quantities.* How much air was needed and what was the best method for distributing the air in the liquid?

(3) *Air sterilisation.* What method was to be used for large quantities of air?

(4) *Agitation.* Would mechanical agitation be necessary in addition to that caused by air bubbling through the medium?

(5) *Materials of construction.* What metals could safely be used for fermentation vessels?

(6) *Pressure vessels.* What steam pressures must be used for sterilisation?

(7) *Cooling methods.* What was the best method for cooling the reactors to keep within the range for successful mould growth?

(8) *Sterilising of plant and medium.* What conditions of time and temperature were required for this to be effective?

(9) *Filtration.* What type of filtration plant was to be used in the final stages of the process?

(10) *Hazards.* Would any special hazards, health problems or nuisances be caused by this new process?

Unpredictable aspects of development

These were the ten principal groups of problems. It will be evident that few of the questions could be answered for the chemical engineer by laboratory work carried out in glass flasks. The men who designed the first plants had to proceed under a great handicap, and their task was made much more difficult by the fact that this new process was loaded on to an already overburdened war-time economy. In many cases, sheer guesses had to be made and equipment used because it was available rather than because it was suitable.

Even when the problems of design in the fermentation stage were overcome, multi-stage extraction plants had to be designed to match fermentation capacity, as well as filling plants to put the sterile finished product into injection bottles for medical use.

Because of the many unknowns, nearly every plant was designed with considerable elasticity, allowances of up to 100% being quite common. Such allowances were very soon made inadequate by the biologists, who produced new strains of the mould which fermented more quickly or produced a higher concentration of penicillin. Almost as soon as a culture was discovered which was twice as good as any previously known, another was found which was twice as good again. This has happened repeatedly, so that the productive capacity of given fermentation plants has increased beyond any allowances a chemical engineer might reasonably have made. There has been a continuous struggle, not only to expand extraction plants to match increased fermentation output, but also to improve the design of extraction equipment to obtain better and better efficiency of recovery of penicillin.

As the output of finished penicillin expanded, an increased number of injection bottles had to be filled. This is just as complex a part of the process as fermentation and recovery, and in relation to the other two sections requires a much bigger labour force and more building space. In practice, just as filling plants began to expand, clinical methods also began to change in favour of much bigger doses in each bottle and this helped in some degree to ease the difficulties of filling. At present in this country over 100 million injection bottles a year are used for penicillin alone, so that a change in output or in clinical practice can not only have an effect on the penicillin manufacturer's flowsheet but can also have a major effect on certain sections of the glass industry.

The pioneer designers should not be criticised because their original flowsheets have been repeatedly thrown out of balance. They could only have provided for what has happened by wild guesses and extravagant expenditure, since the factors concerned were quite outside their control.

Even today the biologist cannot forecast the maximum yield that is likely to be obtained by fermentation, and the clinician cannot forecast the final preferred form of administration.

A balanced flowsheet today may be out of line in a year's time.

Some solutions to these problems

Reverting to the ten problems of fermentation practice, it might be thought, in view of the success which has been achieved, that all the answers to the questions had been found and that their tabulation was merely a matter of recording history. This is not the case, and many of the questions must be asked all over again each time a new antibiotic is to be manufactured. Intensive research is still going on regarding certain aspects and, although good standards of working practice have been established, much still remains to be achieved, particularly in the economic use of services such as air and mechanical power, which form a significant part of total fermentation cost. In the design of equipment suitable for sterile practice much more can be done to ensure a satisfactory margin of safety combined with ease of operation.

Many papers have been published which set out in much detail the results of investigations on aeration and agitation, filtration and extraction, as applied to antibiotic manufacture. It is proposed to discuss here only certain points which have received less attention in published literature, and in which the interplay of microbiology, biochemistry and chemical engineering are of the utmost importance.

Air sterilisation by heat

Heating is by far the most certain method of sterilising air, but it is the most expensive. At 300°C. all living micro-organisms are destroyed in a few seconds, and at temperatures a little higher death is almost instantaneous. A large holding vessel is therefore not required with air at these temperatures, but the air must be cooled before use in the fermenters. An efficient heat exchanger is necessary.

Sterilisation by heat is used in small-scale production units where electrical heating is usually preferred.

Air sterilisation by scrubbing

Some of the earliest penicillin plants employed wet scrubbing methods with only moderate success. The main difficulty lay in wetting the organisms, which are as small as 1 micron.

The most successful method of wet scrubbing has been to use porous elements for passing air into the liquid scrubbing medium. A depth of not more than 2 ft. has been found the best for the porous elements, which can be sintered carbon or metal, fused silica or quartz. There are, however, difficulties in the application of this method which still need to be overcome.

The porous elements must be robust to withstand frequent heat sterilisation. A fractured element which permits large bubbles to pass destroys the efficiency. At present the unit elements available are small in size and a very large number must be used in parallel. This complicates the design, since each element has a critical air velocity which must not be exceeded in order to prevent coalescing of the bubbles on the surface of the element.

Air sterilisation by filtration

This is the simplest and cheapest method and most manufacturers now employ dry filters. Two types of filters are used, one packed with cotton wool or sintered slag wool, the other packed with coarse materials such as a bed of granular carbon.

A fine packing, a minimum depth of 3 in. and a maximum air velocity of 1 ft./sec. provide adequate conditions for the complete removal of micro-organisms from the air. The chief difficulty is to ensure that no air by-passes the filter. The most usual practice with cotton-wool filters is to build the material as a 6-in.-thick roll on a cylindrical wire mesh enclosed in an outer chamber. This can be sterilised by dry steam.

In a coarse filter such as granular carbon it is thought that direct adsorption of the micro-organisms may take place. A bed of this type may be used for many hundreds of hours without re-sterilisation. The usual size of granule is in the range 20 to 40 mesh, although 12 mesh is effective and 30 to 60 mesh is used, but the pressure drop is increased.

Air testing

The real test of efficient operation is the behaviour of the air in large-scale fermentations. Many air-sampling devices have been proposed by different workers, but none is satisfactory because of the limited volume of air which is used. The volume of air in which the presence of one micro-organism must be detected is very large, and full-scale working test is by far the most satisfactory.

Sterilisation of other materials

The normal method for the sterilisation of the materials in the fermentation vessel, before beginning the process, is by steam at a temperature of 120°C. for 30 min. This is essentially a simple matter, but is complicated when precipitates of insoluble compounds are formed in the fermenter. It is important that any crevices and pockets which trap these solids should be avoided in the design of the plant. All pipelines must be arranged to drain completely, and dead lengths of pipe, such as connections to pressure gauges, must be avoided.

Maintenance of sterility

During fermentation every pipe isolated by a valve from the fermenter is maintained under full steam pressure. No non-sterile services, such as plant water, are allowed to remain in direct connection with the vessel.

The biggest sources of danger are valve stems and the shafts of pumps and agitators. Completely satisfactory answers have still to be found to these difficulties. Diaphragm valves are theoretically sound, but in practice it is found that the diaphragm does not withstand the arduous conditions of steam sterilisation.

Sterile liquids used in fermentation are seldom pumped cold, but are usually moved at full sterilisation temperature, a cooler being placed between the pump and the fermentation vessel.

Correspondence

Batch v. Continuous Distillation

To The Editor: Dear Sir,—I should like to congratulate Dr. Ellis on the remarkably full and also lucid exposition of a very complex subject given in his paper under the title 'Batch versus Continuous Distillation' in your Journal, in July and September 1953.

I should also like to accept your very cordial invitation for comments from readers, which comments I enclose.

H. C. Highet,
The Distillers Co. Ltd.,
Research and Development Dept.
Epsom, Surrey.

Here are Mr. Highet's comments:

Dr. Ellis has selected two typical feed compositions

A. 20% phenol, 80% *ortho* cresol
B. 80% phenol, 20% *ortho* cresol.

Consider Case 'A.' A practical procedure would be to use a stripping column to give say 80% phenol in maximum *yield* (for further rectification, as case B) and thus *ortho* cresol in maximum *purity*, *e.g.* 99.5%. Fig. II in Dr. Ellis's paper shows that as x_F decreases (typical stripping operation) and using the same columns, namely 70, 50 and 35 plates, the necessary high *yield* of phenol is only obtained under conditions favouring the continuous still. Attempts to produce high purity *phenol* from such a feed seem to me to be impractical; but should it be necessary, because of limitations of equipment or for other reasons (*e.g.* analytical and laboratory distillation), to obtain the minor component in maximum purity, then the advantage of the continuous still is not so marked. In analytical distillation both high purity and yield are required for each component. Dr. Ellis shows that this requires ample plates (assuming adequate ratio of charge to hold up), again tending to favour the continuous still from the theoretical if not the practical viewpoint.

Dr. Ellis has not given us the no-hold-up equivalent of Figs. IX and XI. In practice I have found hold-up to have a very deleterious effect on batch stripping operations where the more volatile component is also the minor component. Dr. Rose and I are not in agreement on this point, but I think Dr. Rose agrees now that yield and purity must be considered when comparing batch and continuous distillation.

Consider Case 'B.' Practical procedure would involve a rectifying column to give phenol in maximum *purity* (*e.g.* 99.5%) and *ortho* cresol in maximum *yield* but moderate purity (*e.g.* 80% cresol and 20% phenol for further stripping, case A). Again it is the *yield* of minor components, in this case cresol, which is important and which governs the purity of the major component. These conditions obtain in Figs. IV and V of Dr. Ellis's paper; and in spite of the low purity of the cresol the yield of pure phenol will be high (about 94% in the case given) again favouring the continuous still in most cases.

If we adhere to the precept that the major component must be obtained in maximum purity, then the *yield* of minor component must be as high as possible and since it is the minor component its *purity* will have little effect on the *yield* of the major component. If this obtains, which I submit is good distillation practice, the continuous still is theoretically more efficient than the batch still, except in some cases where as Dr. Ellis points out the number of plates is inadequate for the job. The superiority of the continuous still is more marked in stripping than in rectification.

The ultimate choice is usually based strictly on practical grounds, *i.e.* batch stills for analytical purposes or for limited quantities of materials of varying composition. Availability of stills is also a factor. The theoretical viewpoint is however very important, and I can state that theoretical considerations have led me to the rapid solution of an industrial problem relating to the poor performance of a bubble-cap column employed in a stripping operation. I am therefore very grateful to Dr. Ellis for the emphasis he has placed on theory and for the clarity with which he has done so.

Large-Scale Production of LIQUID GASES

A new $3.5-million low-temperature engineering laboratory for the large-scale production of liquefied gases not previously available in sufficient quantity for laboratory and industrial development and testing has been placed in operation by the U.S. Government at Boulder, Colorado. Known as the National Bureau of Standards-Atomic Energy Commission Cryogenic Engineering Laboratory, the new installation consists of a liquid-hydrogen plant and a cryogenic laboratory and is fully equipped for the development and evaluation of low-temperature equipment for use at temperatures near absolute zero. Besides the production of liquid hydrogen and liquid nitrogen in unusually large quantities, the laboratory will undertake research and development directed chiefly toward the improvement of low-temperature equipment.

LIQUEFIED gases, including oxygen, hydrogen and nitrogen, are increasingly being used in industry and national defence and thus it has become necessary to design larger, more convenient and less hazardous equipment for producing and handling them. At liquid-hydrogen temperatures, normally dependable constructional steels become brittle, rubbers lose their elasticity and the mechanical properties of plastics are greatly altered. One of the functions of the new laboratory will be to investigate the structural and other engineering properties of matter at low temperatures and to develop more satisfactory materials and equipment for low-temperature use.

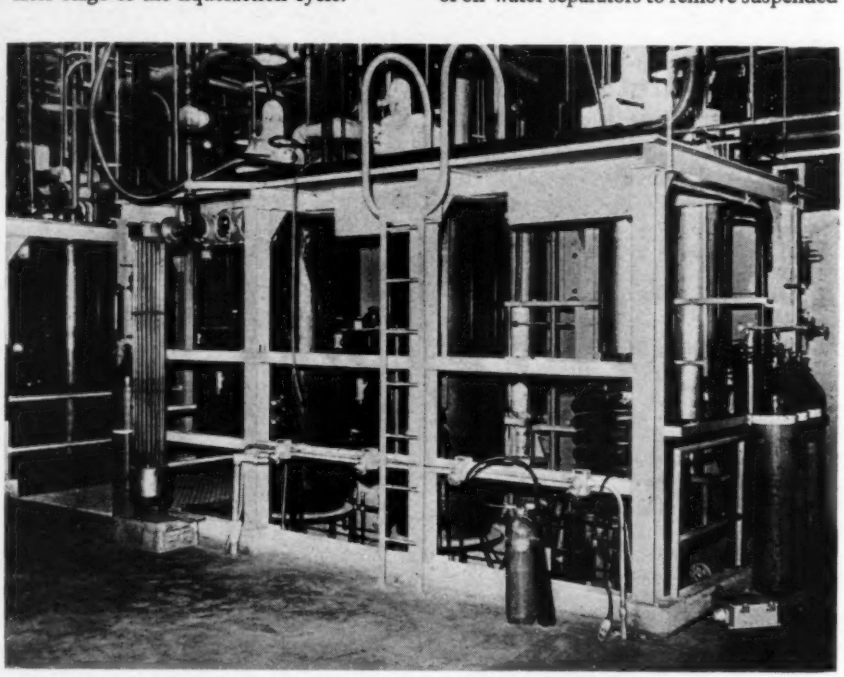
Hydrogen liquefaction

Liquid hydrogen, the lightest of all known liquids, has a density one-fourteenth that of water and boils at 20°C. above absolute zero at atmospheric pressure. Only liquid helium has a lower boiling point. Originally classed by Faraday as a 'permanent gas,' hydrogen has such a low critical temperature (–240°C.) that it was not liquefied until 1898. If at ordinary temperatures (above –80°C.) hydrogen is allowed to expand through a throttling valve without doing external work—the so-called Joule-Thomson expansion—the result is not a cooling effect, as with most other gases, but a heating effect. Evidently, at temperatures above –80°C., known as the inversion temperature, the thermal energy lost by the expanding hydrogen gas (in overcoming the relatively slight forces of molecular attraction between the light-weight hydrogen molecules) is more than balanced by the heat produced as the expanding gas molecules push each other out of the way. Thus, in order to liquefy hydrogen by expansion, it must first be cooled below –80°C. by some other means.

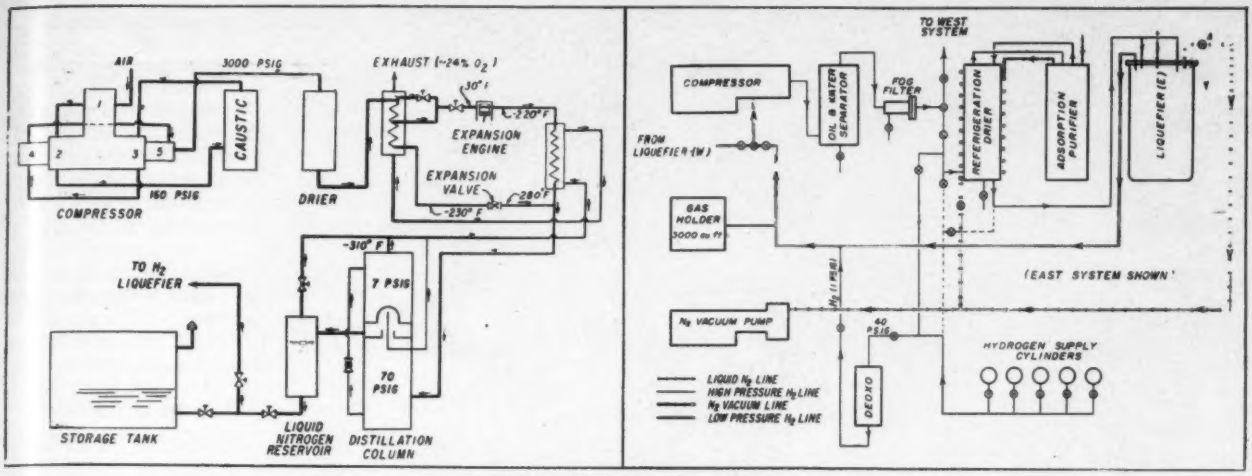
The more hydrogen is cooled below the inversion temperature before expansion the greater is the Joule-Thomson cooling effect. Thus in the Bureau's hydrogen liquefier the hydrogen, following compression, is pre-cooled with liquid nitrogen to as low a temperature as possible before the Joule-Thomson expansion. Liquefaction is then accomplished by allowing the compressed, cooled hydrogen to expand through the Joule-Thomson valve from a pressure of about 1,800 p.s.i. to atmospheric pressure. For maximum efficiency in cooling during the course of the process, extensive use is made of counterflow 'heat exchangers,' in which the high-pressure gas cools as it passes through many turns of coiled tubing surrounded by low-pressure colder gas moving in the opposite direction from a later stage of the liquefaction cycle.

Commercial hydrogen for liquefaction is stored at the plant in mobile and stationary tube banks at a pressure of 2,100 to 2,400 p.s.i. From the storage tubes the gas flows at reduced pressure (25 to 50 p.s.i.) through a tank containing a palladium catalyst, which removes small amounts of oxygen, and then to water-cooled four-stage compressors.

Leaving the compressors at room temperature and a pressure of about 2,000 p.s.i., the gas is next purified by a system of oil-water separators to remove suspended



HYDROGEN. To ensure continuous operation, the hydrogen liquefying and purifying equipment at the new laboratory was constructed entirely in duplicate. This view shows one complete liquefying and purifying system (right of the central passageway) and a portion of the second system (at extreme left). Just right of the passageway is a 1,000-litre metal dewar which surrounds the hydrogen liquefier with its heat exchangers and liquid-nitrogen precooling pot. To the right of the liquefying dewar is a second large dewar which surrounds a silica-gel purifier for removing nitrogen impurity from the compressed hydrogen before liquefaction. To the right of this dewar is an alternate or 'switch' purifier from which the dewar has been removed to expose a large coil (below), containing silica gel, and the heat exchanger above the coil. The tall cylindrical case behind and to the right of the open purifier contains a refrigeration drier, whose function is to remove water in the form of ice from the compressed hydrogen before it reaches the purifier.



Flow diagrams of (left) the nitrogen liquefier and (right) the hydrogen liquefier, at the new laboratory.

liquid, a refrigeration drier to remove water and oil vapour, and an adsorption purifier of silica gel cooled to liquid nitrogen temperature for removal of nitrogen and other gases. After the purified hydrogen has given up some of its heat energy in passing through counterflow heat exchangers, further cooling is attained in the liquid-nitrogen precooler, where the hydrogen passes through coiled tubing immersed in liquid nitrogen boiling at reduced pressure. From the pre-cooler, the gas passes through a final heat exchanger and then through the Joule-Thomson expansion valve, where part of the hydrogen is liquefied by the expansion. Upon leaving the liquefier, the liquid hydrogen is transferred into storage containers through specially designed vacuum-insulated transfer lines, 50 ft. or more in length. It appears likely that these 'pipelines' can be greatly extended if desired.

The hydrogen liquefier

The hydrogen liquefier is unique in that all the heat exchangers as well as the liquid-nitrogen pre-cooler are contained in a single, large, vacuum-insulated, metal dewar having an internal length of $7\frac{1}{2}$ ft. and an internal diameter of $2\frac{1}{2}$ ft. An identical dewar surrounds the silica-gel adsorption purifier. The upper two-thirds of the liquefier dewar contain three heat exchangers and the liquid-nitrogen pre-cooler. These heat exchangers are in parallel and are equipped with adjusting valves which permit distribution of the flow of entering hydrogen between the exchangers in such a way as to obtain maximum efficiency in the cooling process. The lower third of the liquefier dewar serves as the liquid hydrogen collection pot. It also contains the final heat exchanger and the Joule-Thomson expansion valve. The portion of the hydrogen which was cooled but not liquefied in passing through the expansion valve is used as a refrigerant in the final heat exchanger and in one of the initial heat exchangers, while the liquid nitrogen vapour from the pre-cooler is the refrigerant in the other two heat exchangers.

The refrigeration drier and silica-gel adsorption purifier are arranged as a unit. Hydrogen gas enters the drier case at room temperature and passes over heat exchanger tubes where it is cooled by out-flowing gas inside the tubes. The water and oil impurities are condensed out as the gas flows through the drier towards the nitrogen purifier. The gas is finally cooled to liquid nitrogen temperature by passing through a coil immersed in liquid nitrogen in the purifier dewar. At this temperature, nitrogen gas present as an impurity in the hydrogen is readily adsorbed by the silica gel as it flows through the silica-gel coil.

Nitrogen liquefaction

Liquid nitrogen (99.7% pure) for pre-cooling the high-pressure hydrogen in the liquefier is produced in the liquid nitrogen plant, which consists of two complete and independent commercial units for purifying and liquefying the gas. Both units are usually operated simultaneously. Two 11,000-litre insulated storage containers, each having a loss rate of about 1.5% a day, make it possible to maintain a large supply of liquid nitrogen at all times.

For liquefaction at atmospheric pressure nitrogen must be cooled to -196°C. In the new plant, liquefaction is attained a few degrees above this temperature because the pressure of the gas is kept somewhat higher. During the course of the process, nitrogen is separated from ordinary air drawn in from the roof of the plant and is ultimately cooled to liquefaction by a combination of processes involving refrigeration by contact with colder gases flowing back from later stages of the cycle, expansion through a Joule-Thomson valve and through an air-expansion engine, and fractional distillation for removal of oxygen and further liquefaction.

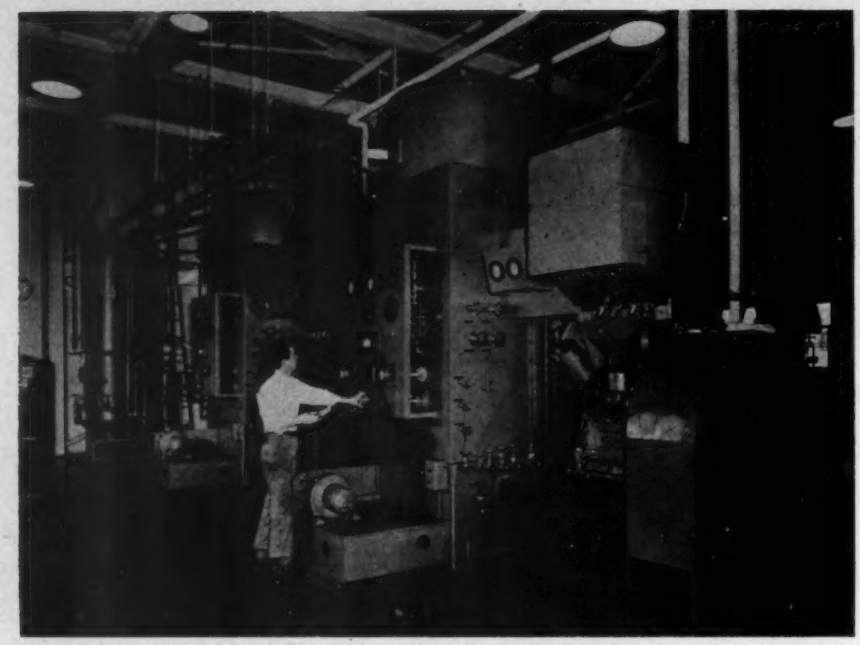
The air from which the nitrogen is to be separated is first compressed to 3,000 p.s.i. in five stages. Between the second and third compression stages it is run through a tank containing a solution of sodium hydroxide for removal of carbon dioxide. After three more compression stages the pressure is brought to 3,000 p.s.i. and the gas flows through a drier containing activated alumina for removal of moisture.

With carbon dioxide and water removed, the gas now consists essentially of nitrogen and oxygen at relatively high pressure. It next enters a heat exchanger, which it leaves by two different lines. One line is connected to the cold end of the heat exchanger—about -145°C.—the other is connected at a point where the temperature is about 0°C. Through this latter line the high-pressure gas enters an expansion engine. Here its pressure is lowered as it does work against reciprocating pistons. The work thus developed is absorbed by an electric generator, and this loss of energy from the gas results in a lowering of its temperature to about -140°C. Further temperature reduction down to the liquefaction point is then obtained by passage through an auxiliary low-pressure heat exchanger.

The line leaving the cold end of the main heat exchanger goes to an expansion valve, where the pressure is reduced to about 5 atm. The temperature is correspondingly reduced to the liquefaction point by the Joule-Thomson cooling effect, causing a fraction of the air to liquefy.

The expansion engine furnishes a sizable amount of refrigeration which indirectly serves to pre-cool the air going to the expansion valve, thus increasing by several times the efficiency of the process. Optimum performance of the system is obtained by regulating the amount of air taken by the expansion engine. This is controlled by the speed of the expansion engine and the time the intake valve is open.

The two streams of partially liquefied



NITROGEN. The liquefaction plant contains two complete liquid-nitrogen generators which separate nitrogen from the air and liquefy it. Within the large cylindrical metal case is a condensation liquefier and liquid-air distillation column which removes oxygen from the liquid-air mixture. To the right of the large column is an air-expansion engine. Air entering the engine at about 0°C. and 3,000 p.s.i. expands to 70 p.s.i. In the course of the expansion, the gas does work against reciprocating pistons, and the energy thus lost results in a lowering of its temperature. The smaller-diameter tank at the extreme left contains activated alumina for removal of moisture from the air at an early stage of the liquefaction cycle.

nitrogen-oxygen mixture join and flow to a distillation column where the liquid nitrogen is separated from the mixture. Refrigeration for condensation of the nitrogen vapour which accumulates in the top of the distillation column is obtained by piping some of the oxygen-rich liquid in the bottom of the column into a jacket surrounding the top of the column. The liquid nitrogen is continuously tapped off into a reservoir and periodically transferred to the large storage tanks.

Liquid *para*-hydrogen

Recently efforts to obtain liquid *para*-hydrogen on a large scale have met with success. Liquid hydrogen as usually produced has a high concentration of the molecular form known as *ortho*-hydrogen, in which the diatomic molecules have the two nuclear spins oriented in the same direction. But the low-temperature equilibrium form of hydrogen is nearly pure *para*-hydrogen, in which the nuclear spins in each molecule are opposed. Thus, ordinary liquid hydrogen slowly changes to the *para* form with an accompanying evolution of heat, and this heat causes a serious loss of liquid hydrogen by evaporation (about 1%/hr. for fresh normal liquid hydrogen). One of the liquefiers at the new Cryogenic laboratory has been equipped with a special catalysing chamber which results in the production of almost pure liquid *para*-hydrogen. This liquid hydrogen, being free of exothermic *ortho-para* conversion, has excellent keeping qualities and can be stored with little loss for many days.

Safety devices

Among the many automatic safety devices in the new laboratory is a hydrogen-gas indicator which continuously samples the air at eight points throughout the plant building. Whenever the hydrogen concentration reaches 10% of the lower explosive limit, this device sounds an alarm and automatically closes all sources of hydrogen. Other safety precautions include conducting floors to eliminate static sparks, a system which measures the concentration of oxygen in the hydrogen with a sensitivity of 2 p.p.m., and means for venting hydrogen to the outdoors in an emergency.

The work at the new laboratory is directed principally toward safer and more convenient means for handling liquid hydrogen and towards increased knowledge of the properties of materials at very low temperatures. Efficient design of low-temperature equipment requires information on the mechanical and heat-conducting properties of many metals, alloys, and plastics over a temperature range extending from room temperature down almost to absolute zero. The Government scientists are now developing apparatus for low-temperature measurement of the tensile and impact behaviour and fatigue properties of these materials.

Recent publications

Controlled volume pumps, made by Milton Roy Co., Philadelphia, U.S.A., are so called because they deliver a controlled volume of liquid which may or may not be in proportion to another metered variable. Proportioning action by any single pump, on the other hand, is achieved by an external control which is not part of the pump itself. A 40-p. pamphlet illustrating and describing these pumps, with specifications and selection data, has been issued by the company. Typical applications in the chemical industry include continuous polymerisation, esterification, nitration, fermentation, hydrogenation of fatty acids and high-pressure synthesis.

Process steam economy. Technical Bulletin No. 18, issued by Spirax-Sarco Ltd., contains many practical hints on steam efficiency. A logical progression is made round the steam circuit, from the boiler house and back again, and attention is drawn to places where improvements in heating performance, plant output, steam economy and processing costs might be made. Principal subjects covered are: 'storekeeping' of steam; combined heat-and-power working; steam quality; steam distribution; thermostatic control; ways of reducing total heat demand; improving heat transfer at condensing surfaces; steam trapping methods; the air-in-steam problem and automatic venting; improved methods of condensate recovery; and flash steam recovery. A final chapter considers the influence of long-standing habits on peak steam loads.

Epoxide resins for surface coatings. A new 45-p. illustrated booklet by Shell Chemicals Ltd. deals with the properties and applications of their *Epikote* resins. A feature is the reference chart of end uses, where suitable formulations are suggested for a number of applications including coatings for domestic appliances such as washing machines, refrigerators, electric mixers and other kitchen equipment, motor-car finishes, can and drum linings, wire enamels, furniture finishes, floor and spar varnishes, and coatings for concrete and chemical plant.

Whilst specific formulations are given in many cases, the booklet is mainly designed to cover general background information on the performance of the different types of coatings which can be made with these resins. For instance, the effect of increasing 'oil length' of the esters on such properties as solubility, hardness, gloss and drying time, etc., is indicated.

The last ten pages of the booklet are devoted to technical data of general value, such as temperature, colour and viscosity conversion charts, and typical constants of a large number of oils and fats. Full details of the properties of a wide range of solvents, latent solvents and diluents are also given.

Chemical and Engineering Research in Australia

Investigations over a vast field of pure and applied science are described in the fourth annual report of the Australian Commonwealth Scientific and Industrial Research Organisation covering the year to June 30, 1952. Here are some extracts of especial interest to chemical engineers and industrial chemists. Extracts from the Organisation's first, second and third annual reports appeared in our September 1950, November 1951 and December 1952 issues.

CHEMICAL ENGINEERING

Utilisation of low-grade coal

THE chemical engineering research programme of the Organisation is at present confined to the complete gasification of Victorian brown coals to produce both synthesis gas and gas of high calorific value suitable for distribution as town gas. Pilot-plant research is being supported by fundamental work on the properties of brown coals and the mechanism and kinetics of the chemical reactions involved in the complete gasification of coal. A new synthesis gas pilot plant employing a fluidised fuel bed has been completed and is now being used in a systematic study of oxygen-steam gasification in this type of system. It is intended that this study will be extended by the construction of a further pilot plant in which gasification will be carried out at pressures up to 20 atm. For this work information is required on the influence of superatmospheric pressures on the rate of the carbon gasification reactions, and the special reactor required for this investigation is now under construction.

Further research has been carried out on the fusion and clinkering properties of brown-coal ash. Particular attention has been given to a study of the mechanism of the initial sintering of ash which may occur at temperatures 400 to 500°C. below the final fusion temperature.

A study of the catalysis of the water-gas shift reaction by which carbon monoxide and steam react to form hydrogen and carbon dioxide during brown coal gasification has shown that carbon surfaces exert practically no influence on this reaction, but that various types of brown-coal ash are very active catalysts. The active constituents in the ash have been identified and a tentative mechanism derived. A theoretical study has been made of the influence of several variables on the reactivity of carbon to steam, oxygen and carbon dioxide. Particular attention has been given to the influence of ash constituents on carbon reactivity.

Research on the production of high-calorific-value gas is concentrated on the investigation of the rate and mechanism of methane formation during pressure gasification of brown coal. To this end it is planned to investigate the reaction of carbon monoxide-hydrogen mixtures with brown coal and brown-coal chars in fluid beds under pressures ranging from 15 to 50 atm. Most of the special high-pressure equipment required for this work is now constructed, and erection of the plant is well advanced. A programme of work on the fluid-bed carbonisation of brown coal has been completed.

Several short-range co-operative research projects undertaken included an investigation of the fluid-bed carbonisation of Leigh Creek sub-bituminous coal, aimed at the production of a low-volatile pulverised fuel for use in an experimental slag dezincing furnace; an investigation of the utilisation of high-moisture raw brown coals for the manufacture of producer gas for firing lime kilns; and an investigation of the suitability of low-ash brown coals for the manufacture of electrode carbon for the aluminium reduction industry.

Advice has been given to a number of organisations on the properties of brown-coal ash, and to one company on the use of wood fuel for producer-gas manufacture.

ATOMIC PHYSICS

Tracer elements investigations

Towards the end of 1951 new arrangements were made for tracer elements work; staff of the organisation previously working the Commonwealth X-ray and Radium Laboratory were transferred to a Tracer Elements Investigations Section. This group is available to assist other divisions and sections, and university and other laboratories, with problems associated with the use of isotopes in non-medical research, to synthesise chemical compounds isotopically labelled and to carry out independent research.

Through the courtesy of Prof. Hartung, temporary laboratory and office space has been provided in the Chemistry School, University of Melbourne. Part of this space has been air-conditioned and equipped as a counting room, allowing radioactive assay work to be kept separate from chemical work.

(*a*) **Radiochemical synthesis and processing.** A simple high-vacuum system for handling small amounts of radioactive gases and vapours is operating on a routine basis. Many simple organic reactions, such as Grignard reactions and lithium aluminium hydride reductions, can now be performed on a millemole scale with this apparatus.

Suitable methods of synthesising labelled compounds that are not readily available overseas are being developed. 'Methoxone' (2-methyl-4-chlorophenoxy-acetic acid), glycerol, oxalic acid, α-ketoglutaric acid and various ketones, α-diketones and α-glycols are all required labelled with C^{14}. A long-chain mercapto-acid labelled with S^{35} is also needed.

A combustion train has been set up for the dry oxidation of organic material containing C^{14} to barium carbonate, and for its subsequent mounting for assay as 'infinitely' thick samples. This is an alternative to the wet oxidation procedure with the Van Sylke-Folch reagent.

Radioactive sources for various users have been prepared on suitable mounts when needed.

(*b*) **Isotope effect on chemical reaction rates.** The C^{14}-isotope effect in the Cannizzaro reaction of C^{14}-formaldehyde has been studied in conjunction with Dr. G. M. Harris, of the University of Melbourne. For this reaction the rate constant for C^{14} molecules was found to be about 6% less than for C^{12} molecules. An effect of similar magnitude has also been found for the reaction of C^{14}-formaldehyde with dimedone. Equations have been derived which express the specific activities of an isotopic reactant and product at any stage of a reaction involving an isotope effect.

Other well-known organic reactions are being studied to see if they exhibit C^{14} isotope effects. The latter have more than a theoretical significance, since they may have an important bearing on the accuracy of a tracer study.

(*c*) **Pilot and assay experiments.** Further assistance has been given to the Irrigation Research Station, Griffith, with the assay of radiosodium, Na^{22}, samples in the study of salt movement in irrigated soil. For this work, and for the assay of weekly radioactive powdered mineral samples, an annular lead castle has been constructed. This method of assay is very convenient for γ-ray emitters, because little time is spent on sample preparation.

The apparatus is at present being used by the Ore-Dressing Laboratory for the assay of monazite-containing mineral fractions.

The penetrating nature of γ-rays enables suitable radioactive sources to be detected by a Geiger counter or other means, through quite large thicknesses of interposed material. Many descriptions have been published of different practical applications of this radioactive method of marking hidden objects, and it is evident that a difficulty often encountered is to obtain an accurate directional location of the hidden source, while keeping its radioactive strength at a reasonably safe (and economical) figure. This problem is being studied for the case of buried sources through moderate thicknesses of soil.

Radiophosphorus, P^{32}, has been applied as a tracer in the Central Experimental Workshops to test the efficiency of the centrifugal drying of washed dried fruit; it has provided a very sensitive means of measuring the minute amount of water retained on the surface of the fruit.

(*d*) **Information.** Information on equipment and methods for radioactive tracer work, and on various applications, has been supplied to local and overseas workers. Most of the stock of lead castles made in the Central Experimental Workshops has now been disposed of, and negotiations are being made for a commercial firm to undertake their further manufacture to C.S.I.R.O. designs.

INDUSTRIAL CHEMISTRY

The Organisation's chemical research is undertaken mainly by the Division of Industrial Chemistry. E. A. Guggenheim, F.R.S., Professor of Chemistry in the University of Reading, went to Australia and spent about two months with the Division. An acknowledged authority on thermodynamics, he has wide interests in theoretical chemistry and was able to make a substantial contribution to many projects.

Minerals utilisation

The research programme of this section is based on the development of new methods of processing minerals and the fundamental study of the chemistry of their constituents in order to extend the range of their derivatives and their industrial applications.

(*a*) **Rare earth minerals.** Work on the separation of thorium, cerium and the cerium group of lanthanons referred to in the 1950-51 report has continued. Separations based on fractional crystallisation, or partial precipitation, of mixtures have been supplemented by processes using ion-exchange columns for preferential absorption of organic complexes of the lanthanons, and solubility studies in non-aqueous solvents were also made. Investigations on the chemistry of thorium compounds have been continued particularly with regard to the carbides and iodides which are of importance in the production of the pure metal. Methods of general applicability for the isolation and purification of thorium compounds from crude rare earth mixtures were almost completed and studies on the complex potassium fluorides of thorium were concluded. During the year satisfactory small-scale production of cerium oxide optical polishing powder was reported by one Australian company normally engaged in mineral production from beach sands. The process used was that developed by the section in its earlier work on the applied chemistry of the lanthanon group.

(*b*) **Zirconium minerals.** The eastern Australian beach sand deposits of zircon are among the most extensive in the world. Zircon is a rich ore of the increasingly important metal, zirconium, and a source of a wide range of non-metallic derivatives relating to industrial refractories, ceramics, etc. Developmental work on the patented process for the separation of zirconium from the closely related element, hafnium, reported in 1950-51, has continued. Satisfactory progress has been made and a notable feature of the preferred method is that both the zirconium and hafnium, when resolved, are in a form which admits of their conversion to the respective metals without further modification.

(*c*) **Titanium minerals.** Apart from the problems of processing the titaniferous uranium ores of South Australia, the section has been chiefly concerned with the extensive deposits of high-grade rutile which are such a remarkable feature of the heavy mineral sands of the eastern Australian beaches. Apart from its utilisation as such, in electrical welding-rod fluxes, this rutile offers opportunities for the production of many titanium derivatives which are less conveniently or less economically derived from lower-grade minerals such as ilmenite. For some time past a series of products derived from titanium tetrachloride has been under investigation. The study of the properties of titanium nitride has been continued. This material when suitably bonded shows very promising qualities as a hard 'metal' in the same sense that tungsten carbide is so described. As in all such products it is necessary to compromise between the two related qualities of hardness and brittleness. To this end it has been necessary to study many interrelated factors, such as appropriate bonding metals, compacting pressures, heat treatment, etc. Recent results from this work suggest that a useful industrial product may be devised.

(*d*) **Manganese minerals and graphite.** Manganese oxide and graphite are both required for electrical dry-cell manufacture and, although both minerals occur in Australia, it was previously considered necessary to use imported materials. Work was first directed to elucidating the obscure factors which determine whether any particular manganese oxide is suitable for use as a depolariser. It was necessary to investigate both fundamental and applied aspects of dry-cell chemistry, and the knowledge gained has been of use to Australian manufacturers who have collaborated throughout. It has been shown that traditional objections to certain impurities in the minerals are unfounded, and means for the removal of deleterious impurities have been devised. The investigation has shown quite clearly that certain Australian minerals are in no way inferior to accepted overseas materials. The original objectives of the project have now been achieved, but work is being continued on certain fundamental aspects likely to be of importance in other fields.

X-ray diffraction studies relating crystal structure of various manganese oxides with their chemical reactivity have been extended to analyses, now completed, of the crystal structures of the manganese minerals cryptomelane, lithiophorite and psilomelane; an analysis of the structure of chalcophanite is proceeding. Apart from the studies of the behaviour of South Australian graphite in electrical dry cells referred to above, work has been continued on the lamellar complexes which, under certain conditions, graphite forms with some metallic chlorides and related salts. The number of such compounds has been notably increased in the current period and attention has been given to the possibility of their industrial use in the selective separation of the vapours of certain mixed metallic chlorides.

(*e*) **Uranium ores.** The chemical processing of two Australian uranium ores has been investigated. In each case the object of the work has been to devise a method for the chemical extraction and isolation of the uranium as a salt or oxide from either mineral concentrates or crude ore. The titaniferous iron ore, containing davidite, from Radium Hill, South Australia, has been processed with good recovery values by leaching with acids and solvent extraction. The process, which was taken to pilot-plant stage, allowed the recovery of certain by-products as well as uranium.

Attention is now concentrated on methods for processing a different class of uranium ore from Rum Jungle, in the Northern Territory. Laboratory-scale tests indicate that this ore is likely to prove amenable to the treatment devised without prior mechanical concentration and may be expected to yield high recoveries of its useful components.

Cement and ceramics

The programme covers a wide range of investigations on problems connected with portland cement and general ceramics; it includes investigations on pozzolanic cements and on various types of refractories, and a survey of Australia's resources of ceramic clays.

The section has received both practical and financial assistance from the Cement and Concrete Association of Australia and from the National Gas Association of Australia.

(*a*) **Cement-aggregate reaction.** Further papers have been published on the general mechanism of this type of reaction, which sometimes occurs between cement and certain types of sand or stone used as aggregate in concrete. It can lead to expansion, cracking and subsequent deterioration in concrete structures. Most of the laboratory observations which have previously been made on this problem have been based on the behaviour of mortar bars containing high-alkali cement and reactive aggregate of fine grain-size. Such mixes, with the addition of coarse aggregate, are now being studied in order to obtain a more accurate prediction of the behaviour of concretes used in normal constructional practice from the results obtained in a standard testing procedure. Attention is being given to methods for restoring concrete which has cracked and for the prevention of further damage by subsequent weathering. The rate of movement of alkalis in cement pastes has also been the subject of a laboratory investigation which has shown that potassium tends to diffuse more rapidly than sodium. The actual rates at which potassium and sodium hydroxides react with silica are, however, very similar and, in order to gain further knowledge of this reaction, a comparison has been made with the behaviour of lithium hydroxide, which itself reacts more slowly and is known also to have an inhibiting effect on the reactions of the other alkali hydroxides. A radioactive tracer technique is also being used to study the reactivity of different types of silica. Arising from this general investigation is a study of the mechanism of expansion cracking in cement paste, mortar and concrete. Apparatus is also being developed for a study of shrinkage cracking.

(*b*) **Pozzolanas.** These are materials which, either in the natural condition or after heat treatment, possess cementitious properties when finely ground and mixed with lime. They are sometimes used to improve the durability of concrete and to give increased resistance to reaction between cement and aggregate. A description of methods which have been developed for evaluating them is now in the press. The study of the properties of a variety of pozzolanic materials, mostly of Australian origin, has been continued, with particular reference to mortars made from lime and pozzolana and from portand cement-pozzolana blends.

(*c*) **Surface-active agents in cement and concrete.** The durability of concrete may be improved by the use of surface-active agents to promote air entrainment. Current studies include the effect of soluble materials derived from hydrating cement on the effectiveness of various agents and the relationship between sedimentation behaviour and entraining effectiveness.

(*d*) **Mineralogical investigations.** Phase equilibria of silicate systems at high temperatures are being investigated in order to elucidate the occurrence of sodium in cement clinker and of zinc in lead blast-furnace slags. A similar study of basic steel slags has been completed. Equilibrium relationships of certain thorium compounds have been determination in collaboration with the Minerals Utilisation Section; an account of this work is now in the press. Mineralogical investigations have also been made on aluminous and chrome-magnesite refractories which have been attacked by alkalis, on the structure of pozzolanic mortars, on slags and on concrete aggregates.

(*e*) **Cement-kiln refractories.** Bricks of stabilised cement clinkers have been made and burned successfully, and the properties of these refractories are being examined. Work on the production of high-alumina refractories from Australian materials has been begun. The study of factors affecting the durability of kiln linings has been continued. Chrome-magnesite and sillimanite bricks attacked by alkalis derived from the raw materials of cement have been examined and the nature of the deterioration has been revealed. Changes in the cross-sectional dimensions of a cement-kiln caused by heating and rotation are being measured in order to study the stress conditions in the lining.

(*f*) **Gas-works refractories.** A critical study of the physical properties of refractories used in gas retorts in Australia has been published. An investigation of the factors limiting the durability of gas-works refractories under conditions of heavy steaming has continued. A retort so affected has been dismantled and photographed and appropriate specimens collected. Work continues on simulating in the laboratory the conditions in an operating retort.

(*g*) **Clay resources survey.** An extensive account of the ceramic and refractory clays of South Australia has been published. A similar study of Western Australian clays has continued and the laboratory investigations are nearly complete. The examination of samples from Queensland has begun. These samples are being acquired through the collaboration of the Queensland Geological Survey. A critical examination of the methods used to determine the modulus of rupture, base-exchange capacity and dye-adsorption index of clays in this survey has improved the reliability of the values obtained. A thorough examination of the factors involved in dye adsorption may provide a rapid and simple technique for assessing the specific area and activity of clay surfaces, which are of great importance not only in ceramics but in most industrial applications of clays.

(*h*) **Clay mineralogy.** The recent completion of X-ray diffraction equipment has permitted a start to be made on the characterisation of indigenous clays according to their mineral content. X-ray techniques are also being applied to a study of the configurations taken up by water molecules associated with the active surfaces of clay minerals. Work of this kind is aimed at increasing fundamental knowledge of the properties of clay-water systems, and has ultimate application in fields such as agriculture, construction engineering and oil-well drilling as well as ceramics. Direct evidence of the surface-water relationships is difficult to obtain because of the very small grain-size of the clays. A new approach has been initiated by a structural study of vermiculite, a mineral unique in that it forms in clays and also occurs elsewhere as flakes large enough to be investigated by single-crystal X-ray techniques. It is closely related structurally to the important clay mineral, montmorillonite, and, like the latter, consists of complex aluminosilicate layers interleaved with layers of water molecules and carrying exchangeable ions. Evidence of precise orientation of the water molecules and exchangeable ions with respect to the silicate surfaces has been obtained.

(*i*) **Silica investigations.** Microcrystalline silica (flint) is an important constituent of certain English whiteware and has some advantage over quartz, particularly in producing a body resistant to crazing. Efforts are being made to locate Australian sources of supply. A study, more detailed than any previous ones, of the physical properties of both raw and calcined microcrystalline silica has been published. A study of the physical properties of South African silcrete bricks shows that they are of high quality. Australian quartzites, including those of the silcrete type, are being examined with the object of producing silica bricks of a similar standard.

(*j*) **Whiteware and related investigations.** Studies of whiteware bodies have revealed new points of interest concerning their porosities and microstructures. The results are being prepared for publication. For the first time a detailed investigation has been made of the deformation of plastic clay in which the orientation of the clay-mineral crystals is known. An account of this work is in the press. Investigation of the fusion of granitic materials used as fluxes has been specially directed to the collection of gases evolved which may cause ' bloating ' in ceramic bodies.

(*k*) **Saggar investigations.** Saggars developed from South Australian and other materials continue to give good service under industrial conditions. An account of the investigation has been published; it deals also with saggars of improved type which do not require a high initial firing temperature.

(*l*) **Special ceramics.** Titanate dielectrics have been speci-

ally prepared in the section and are giving satisfactory service in equipment being operated by another Government research laboratory.

(*m*) **Foundry sands.** Investigation of organic binding materials for sand cores and of clays for use in blending synthetic moulding sand mixtures has continued.

Physical chemistry

(*a*) **Separation of antibiotics.** There has long been a need for an efficient process analogous to fractional distillation for the separation of the components of certain types of complex mixtures. It has been evident, for example, in the large-scale production of the antibiotics such as penicillin and streptomycin. Attempts have been made to develop a new process based on the fractional adsorption of the active components on solid adsorbents, and applications have been made for patents covering the process. The principle of the method is a countercurrent extraction in which a specially prepared solid on which the antibiotic is adsorbed descends against a rising column of liquid (reflux) which removes the contaminants. A *p*H gradient in the adsorption columns corresponds to the temperature gradient of a distillation column.

It may be operated continuously or used, like a batch distillation, to produce several products during the one treatment and it is economical of both adsorbent and solvent. Some engineering problems have yet to be solved before the process can be regarded as suitable for industrial use. In the course of this work a new adsorbent was developed which may have a wide use for industrial processes because of its cheapness and reproducibility. The equipment which was developed for the above process is also being used in a new process for the continuous softening of water by ion-exchange resins.

(*b*) **Properties of fluids.** Work has been continued to obtain accurate data concerning the physical properties of fluids and the manner in which they are influenced by change of temperature and pressure. To obtain such data and develop methods of predicting the behaviour of fluids under a variety of conditions is of considerable importance in practical problems of chemical engineering design. The second virial coefficients of silane, silicon tetrafluoride and ethylene have been determined with a high degree of precision. These coefficients indicate the magnitude of the departure of certain properties of a gas from those which would be exhibited by a perfect gas.

There is considerable discrepancy between the behaviour of certain light gases, such as hydrogen and helium, at high pressure and that expected on the basis of current theory. A new theory based on the quantisation of the motion of these gases has been developed which has enabled their behaviour to be predicted closely.

Certain problems have arisen during the year which have involved the measurement of dielectric losses of methyl fluoride and ethylene above their critical points, and also the variation of electrical conductivity of ammonia solutions with pressure.

Thermodynamic data are also being obtained on pure liquids and binary mixtures. Equilibrium data have been determined experimentally for the systems benzene-*n*-heptane and their vapours, and *n*-hexane-chlorobenzene and their vapours. Precision equipment is being made for determining the heat of mixing of two liquids.

A theory has been developed from which the properties of mixtures containing alcohols can be predicted. Such mixtures are of considerable industrial importance, but their complexity has hitherto prevented detailed theoretical treatment.

(*c*) **Heterogeneous catalysis.** The adsorbent materials widely used as catalysts in the chemical industries to accelerate chemical reactions are usually fine powders compressed into granules. The physical structure of the catalyst is an important factor in determining its efficiency and, if the mechanism governing the rate of entry of gases into the granules can be established, it becomes possible to calculate the most suitable fineness of powder and size of granule. The rate of entry of the gases has been determined by measuring the transient rates of Knudsen flow and adsorption of water, ethanol and isopropanol vapours in beds of alumina and thoria during periods of time as small as 1 msec. The measured rates have a negative temperature coefficient, are first order with respect to pressure, and certain of them are second order with respect to the amount of adsorbent. The adsorption of gases on metals has been treated theoretically and the results have been used to suggest the mechanism of the Fischer-Tropsch process by which carbon monoxide is hydrogenated to produce liquid fuels.

(*d*) **Fine particles.** The separation of finely divided solids from gas or liquid suspensions is a problem common to many industrial processes. Experimental and theoretical studies have shown that the sedimentation of particles in suspensions is closely correlated with the flow of liquids in filter cakes and can be used as a basis for estimating the efficiency of filtration and improving the design of filters and filtration techniques. The separation of fine particles from gas streams has also been investigated and a new type of centrifugal separator has been developed which will effectively separate particles as small as 2 microns. Some field tests under industrial conditions have confirmed the results obtained in the laboratory. For this work a new type of thermal precipitator was developed for the quantitative measurement of concentrations of fumes.

(*e*) **Condensation of steam.** It is known that the efficiency of steam condensers can be greatly improved if the steam can be caused to condense on a suitably treated cooling surface as droplets rather than as a continuous film. No successful industrial unit has yet been operated on this principle and little is known of the way in which the droplets are formed, the most suitable agents for making the surface hydrophobic (water-repellent) or the conditions under which this surface can be maintained. Equipment has been built to determine the efficiency of agents causing the drop-wise condensation, and the calorimetric heats of immersion of several hydrophobic solids have been measured.

ORGANIC CHEMISTRY

Wool and sugar-cane waxes and allied aliphatic compounds of high molecular weight, plant alkaloids and phenol formaldehyde plastics have continued to be the main interests of this section. The work on plant alkaloids has been chiefly directed to the chemical examination of plants poisonous to stock. The service provided for the microanalysis of organic compounds has continued to be widely used by industry and the universities. As the demand for analyses increases, it is hoped to include additional methods of analysis in the more satisfactory continuous operation. The new direct determination for oxygen is now on this basis.

Sugar-cane wax

This wax is potentially available in considerable quantities in the filter muds from sugar-cane mills. Although an industrially useful wax can be produced after bleaching and refining, attempts have been made to prepare a number of derived waxes which should be of greater value to the wax industry, using as a starting material the mixture of long-chain fatty acids obtained by bleaching a vacuum-refined wax. The difficulties, mentioned in the previous annual report, encountered in converting this wax into a series of ester waxes have been overcome and they can now be readily produced. Representatives of several chemical groups have been prepared; mainly the esters and amides of polyfunctional alcohols and amines. Some of these derived waxes show improved properties compared with the parent acids. Their physical properties and their behaviour in solutions and in gels are being examined to discover their usefulness and, if possible, to obtain information on the relation between their physical properties and chemical structure. In the patent literature many solvents have been claimed as suitable for the separation from the crude cane wax of a product resembling the original cuticle wax. Accurate measurements have been made of the solubility of sugar-cane wax in a range of these solvents, and the results indicate that acetone, ethanol and *iso*propanol are likely to be most suitable for a process of solvent refining.

Ore dressing

Work done at Melbourne is recorded in 23 reports which refer to ores of gold, tin, tungsten, cobalt-nickel, copper-lead-zinc, antimony, bismuth, gypsum and pyrochlore, and to lead furnace slags, beach sands, a filler for plastic products, brown coal and lucerne seed.

The application of magnetic and electrostatic separation to the concentration of pyrochlore from an ore from Uganda was studied.

The application of sink-float and table concentration processes for the beneficiation of dune gypsum deposits in South Australia was investigated.

Attempts were made to develop leaching processes for recovery of bismuth from a Tennant Creek (Northern Territory) ore, and of cobalt and nickel from an ore from New Caledonia.

A polarographic method for determination of dissolved oxygen in cyanidation and flotation plant pulps is being developed; this technique could be useful for testing and control in many ore treatment plants.

The work at Kalgoorlie, in addition to 197 free assays or analyses, resulted in the issue of 45 reports of which 23 involved metallurgical investigation, including three pilot-plant trials. Most were concerned with gold ores and tailings and others dealt with recovery of scheelite and lead and zinc concentrates, beneficiation of limestone and determination of characteristics of sand deposits.

During the year a considerable number of inquiries from the mining industry were answered by the laboratory staff.

Flotation

The study of the flotation process has been continued by the Physical Chemistry Section of the Division of Industrial Chemistry. When the tin mineral cassiterite exists in a finely disseminated form in an ore, substantial losses occur when it is concentrated by conventional gravity methods. Two such ores, one from Ardlethan, New South Wales, and one from Maranboy, Tasmania, have been subjected to preliminary flotation tests using a process developed a few years ago in this laboratory. The Ardlethan ore, because of its high content of topaz, is not amenable to treatment by this process, but the tests on the Maranboy ore have given encouraging results and it is being investigated further.

The study of the process by which air bubbles and mineral particles become attached in a flotation cell has been continued. Since this process determines the capacity of a flotation cell, anything that can be done to increase the frequency of adhesion consequent on collision of air bubbles and mineral particles must have a direct bearing on the cost of treatment of the ore. It has been found that, as the bubbles approach closely to the mineral surface, the water between them flows away according to normal hydrodynamic laws, but at a certain critical distance the residual water film becomes unstable and, with its rupture, adhesion between bubble and particle occurs. The thickness and hence the time required for rupture of this film depends on the degree to which the surface has been rendered hydrophobic (water-repellent). A closely related problem is the rupture of the thin liquid films comprising a foam. A new theory has been proposed which attributes a dominant role to the flow of liquid in the film under the influence of surface tension gradients across the film.

Titanium and its alloys

The Physical Metallurgy Section has been investigating the reaction of titanium and its alloys with air. This is of technological importance, since the high temperatures at which alloys are likely to be employed and the hot working in fabrication will result in surface hardening from reaction with both oxygen and nitrogen.

The rate of absorption of oxygen has been measured under controlled conditions of temperature and pressure. Further, the structure and composition of the resulting scales have been determined by X-ray, microscopic and chemical techniques.

The rate of absorption of oxygen at 760 mm. pressure between 600 and 1,000°C. follows an approximately parabolic law.

It has also been found possible to melt titanium dioxide in the argon arc furnace. By diluting this with excess titanium various melts covering the composition TiO, Ti_2O_3 and TiO_2 have been made and studied.

Carbon Dioxide by Molasses Fermentation

SPECIAL attention is being given in East Germany to the possibility of covering the greater part of the demand for carbon dioxide by the molasses fermentation process and thus saving considerable quantities of fuel. A prototype of a type of plant for which high hopes are entertained was recently inaugurated on the basis of molasses fermentation at a large distillery. The hourly output of liquefied carbon dioxide is 270 lb.

The crude gas coming from the fermenting vats (from which the air had first been purged) is led through a water scrubber to absorb any entrained alcohol before entering the suction line of the liquefying compressor. The latter is also connected through a by-pass to a gasholder for storage of a reserve of gas as a precaution against interruption of crude gas from the distillery operations.

A pressure-equalising vessel is inserted in the suction line of the low-pressure stage of the compressor to even out the high gas velocities (and resultant pressure-drop immediately in front of the suction port) consequent upon the intermittent running of the low-pressure piston. This vessel can also trap any water carried along from the scrubber.

In the low-pressure section of the vertical three-stage liquefying compressor the carbon dioxide is now compressed to 3.2-atm. gauge before passing through a cooling coil immersed in the water tank of the compressor where its heat of compression is abstracted and where condensation of part of the entrained water vapour also takes place. The latter is collected in a separator in front of the low-pressure stage. The compressed gas is now passed through an active carbon adsorber which removes the fermentation flavours and odours. Water is next removed in a silica gel adsorber. Provision is made for regeneration of both types of purifiers (see below).

The purified and dried gas is now ready to enter the medium-pressure stage of the compressor where it is compressed to 17-atm. gauge and the heat of compression is again taken up in the water tank. Finally the liquefaction pressure of 70 atm. is reached in the high-pressure stage. The compressor is fitted with self-lubricating carbon piston rings to prevent contamination of the carbon dioxide with oil.

Superheat and heat of vaporisation is removed in a condenser where the actual liquefaction takes place. Filling into commercial cylinders or tank waggons can then be undertaken at the filling stations operated by the distillery.

Regeneration of purifiers

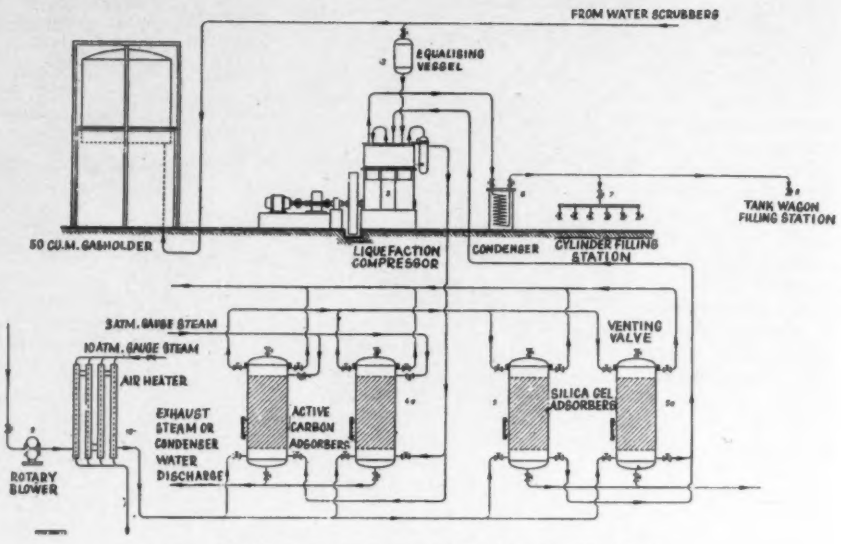
Whilst one set of purifiers (*e.g.* 3 and 4 in the diagram) is in operation, another set will be undergoing regeneration. Steam at 140°C. (3-atm. gauge) is passed in countercurrent through the active carbon tower which will be ready for operation again after cooling and air-drying. The silica gel tower is regenerated by a current of hot air (130°C.), again in countercurrent to the normal direction of flow of the carbon dioxide. It should be noted that the carbon dioxide enters the active carbon column at the bottom, so that the regenerative steaming is effected in a downward direction, whereas in the silica gel column the directions are reversed. The rotary blower 9 is used for forcing cold or hot air, as required, through the columns.

The plant has been operating at the Schönebeck molasses distillery for over

a year and the pure carbon dioxide product has been made available to the food industry. Regular analytical control shows that the product is of high purity, fully comparable to the gas derived from natural wells or from coke.

A striking difference between a distillery-based carbon dioxide plant and a coke-based plant is the much lower weight of the former installation (13.8 tons, compared with 95 tons for a coke-based plant of equal output).

The encouraging performance of the prototype plant has raised hopes of the feasibility of centralisation of the whole East German carbon dioxide industry (solid as well as liquid) at a few distilleries, in which connection the great fermentation plant at Dessau is mentioned as being one of the most attractive for such a project. It is planned to deliver carbon dioxide in bulk to a chain of bottling stations throughout the country in order to limit the long-distance transport of the heavy steel cylinders to a minimum.—E. Kuntzsch, *Chemische Technik*, 1953, 5, (10), 587-590.



Flow diagram of a fermentation carbon dioxide plant.

Enrichment of Industrial Gases by Methane Synthesis

THE catalytic synthesis of methane from the carbon monoxide and hydrogen contained in water gas and other industrial gases has long been known as a possible process for the enrichment of such gases to the calorific value of town gas. A new publication* describes work carried out at the Fuel Research Station, Greenwich, with the object of developing a process suitable for large-scale operation. Such a process could be applied to water gas or producer gas made from coal or coke by existing processes. It could also form the final stage in the production of town gas by the complete gasification of low-grade fuels.

The work was begun in 1939, with the co-operation of the gas industry, with the object of making gas from which liquid methane could be produced as a fuel for road transport. After the war emphasis was transferred to the possibilities of producing town gas from water gas without the use of imported oil. The report describes experiments with both laboratory-scale apparatus and small pilot plant.

The major problem encountered was the effective dissipation of the heat of reaction, since it was found that excessive formation of carbon occurred if the temperature of the catalyst was allowed to rise above about 500°C. Two methods of removing the heat of reaction were tested on the intermediate scale, in plant processing 200 cu. ft./hr. of water gas. In the first method a close-packed bed of catalyst was used, and the bed was subdivided into narrow cells surrounded with a boiling liquid at a suitable temperature. In the second method the catalyst was contained in a perforated sheet-metal cylinder, while the gas passed along the annular space between this cylinder and a cooled wall, and so had access to the catalyst only through the holes in the cylinder.

Both methods gave satisfactory results on the intermediate scale, the total yield of methane per lb. of catalyst used being 2,000 to 3,000 lb., but the second method showed greater promise as a basis for the construction of full-scale plant processing 10,000 to 40,000 cu. ft./hr. of gas. It had the advantage of virtually eliminating the formation of carbon when water gas was used, whereas with liquid-jacketed close-packed beds carbon was formed on the catalyst if the volume ratio of hydrogen to carbon monoxide in the process gas was less than 1.5 : 1.

Intermediate-scale plant designed to operate with continuous replacement of exhausted catalyst gave satisfactory performance.

Methods of construction have been recommended for full-scale plant to process 20,000 cu. ft/hr. of water gas; about 100 reaction tubes of the form used in the intermediate-scale tests would be enclosed in a single cylindrical water jacket 12 ft. high and about 2 ft. in diameter, in which steam would be generated. The purification of the process gas would be carried out by means of alkaline iron oxide at about 200°C., with a space velocity of 200/hr. About 100 cu. ft. of this material would be required, and the purifier could be a cylindrical vessel about 12 ft. high and 3 ft. in diameter. The temperature could be maintained by preheating the process gas by heat exchange with the enriched gas leaving the methane synthesis unit.

The investigation has been concerned only with enrichment of gas at atmospheric pressure. Early experiments showed that the performance of typical catalysts at higher pressures was broadly similar to that at atmospheric pressure, but no special advantages were found and the formation of carbon appeared to be more rapid. It is probable that if methane synthesis at pressures of several atmospheres were desirable (*e.g.* to enrich gas generated under pressure, or to facilitate subsequent removal of carbon dioxide, or for distribution of gas under pressure) plant of the type suggested would function satisfactorily and the formation of carbon could be controlled.

The intermediate-scale plant has been operated with water-jacket pressures up to 300 p.s.i., generating steam at these pressures. Since the jacket temperature had little effect on the performance, full-scale plant could probably operate with higher jacket pressures if this were found desirable.

**Catalytic Enrichment of Industrial Gases by the Synthesis of Methane*, H.M.S.O. 2s. net.

Welded chemical plant. Three new illustrated publications have been issued by the A.P.V. Co. Ltd. One, a 20-page booklet, describes the organisation of the company's factories and enumerates the types of processing plant fabricated. Specifications for aluminium alloys and stainless steels are tabulated. The second publication is an eight-page brochure dealing with the *Bowser* beer filter. Finally, there is a pamphlet giving details of the company's standard range of stainless-steel reaction vessels.

Canadian Fertiliser Manufacturers Build Another Plant

Producing over half a million tons of chemical fertilisers a year, British Columbia's huge mining, smelting and chemical concern, the Consolidated Mining & Smelting Co. of Canada Ltd. (Cominco), is now completing the construction of another ammonium phosphate fertiliser plant costing $9 million which will have a capacity of 70,000 tons annually. Here is a description of the new plant in the perspective of the company's other fertiliser operations.

COMINCO'S chemical and fertiliser operations stem from the presence of sulphur in the orebody of their principal mine, the Sullivan Mine. This orebody consists, in the main, of iron, lead and zinc sulphides and the elimination of sulphur was a necessary step in the refining and metallurgical processes.

How to deal with this sulphur by-product was a problem which became more and more urgent as the scale of operations at the smelting plant rapidly increased. In 1926, it was decided to use the excess of sulphuric acid over the plant requirements to make fertilisers, and, in 1930, a $10 million manufacturing project was begun at Trail, B.C. This involved the construction of an ammonia unit consisting of an electrolytic hydrogen plant, a liquid air plant for nitrogen manufacture and an ammonia synthesis plant; phosphoric acid and phosphate fertiliser plants; an ammonium sulphate plant; storage, office, shop and other facilities. In 1946, plants built and operated at Trail and Turner Siding, near Calgary, by Cominco for the Government during the war on a non-profit basis for the manufacture of ammonium nitrate were bought by the company. This purchase increased Cominco's investments in the chemical industry to about $25 million. The plant now under construction is at Kimberley, B.C.

Four main kinds of fertiliser are produced by Cominco; ammonium sulphate at 450 tons a day, ammonium nitrate (*Nitraprills*) at 550 tons a day, ammonium phosphate (16-20-0) at 300 tons a day and ammonium phosphate (11-48-0) at 350 tons a day.

Ammonia synthesis

Common to all four kinds of fertilisers is ammonia. The ammonia synthesis plants at Trail are of standard design and have a daily capacity of 250 tons. Nitrogen is produced in standard Claude liquid air units, each having a daily capacity of 46 tons of nitrogen. First, the air is compressed to about 275 p.s.i. and, after being purified and dried, it is liquefied at about −184°C. Nitrogen is first drawn off as the liquid air is warmed into a gaseous state, the oxygen, later recovered with additional heating, being collected for use in such operations as oxy-acetylene welding and as enrichment of air supply to certain furnaces.

The roaster building (*left*) and the acid plant (*right*) at the Kimberley, British Columbia, fertiliser factory. Iron sulphide tailings are roasted in a suspension-type roaster, and the gas produced is converted to sulphuric acid.

Hydrogen is produced by the electrolysis of water in 3,215 Trail-type cells. D.C. power is obtained by means of three 10,000 amp., 670 volt, and seven 10,000 amp., 830 volt mercury arc rectifiers. The electrolyte is a 28% caustic potash solution. The electrolytic cells are very efficient and have been improved by the company's engineers. Indeed, the hydrogen plant at Trail is one of the outstanding Cominco installations.

Both the hydrogen and the nitrogen produced have a purity of 99.9%.

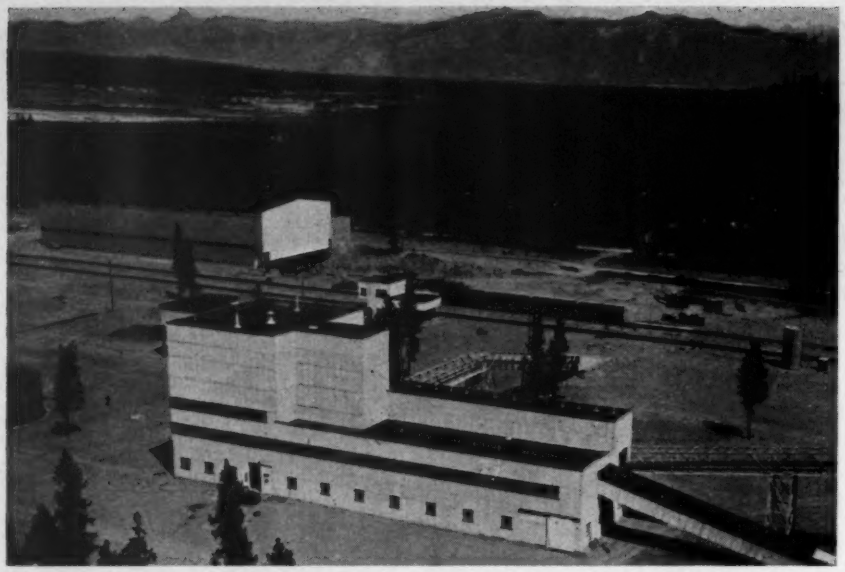
A coke reduction process is used in another plant to produce a gas mixture suitable for ammonia synthesis. This process provides one-quarter of the total feed to the ammonia synthesis units.

In the ammonia synthesis units the nitrogen and hydrogen gases, mixed in the proportion of one to three by volume, are compressed to about 250 atmospheres in six-stage compressors, after which the mixture is circulated through four synthesis columns containing an iron base catalyst self-maintained at a temperature of 400 to 500°C. Only about 15% of the mixed gases combine to form ammonia in one circulation through a catalyst column. The circuit is thus a continuous one, the uncombined gases being mixed with fresh gases and re-passed through the columns.

The ammonia produced is partly condensed into a liquid in a cooling cycle and is stored as liquid in hortonspheres. The balance is transported straight to the consuming plants, part being used for the direct production of fertilisers. The remainder is dissolved in water to form a concentrated aqua ammonia, which is the absorbent for sulphur dioxide taken from the sintering machines in the lead smelter. From the suspension roasters in the zinc plant, gas containing up to 7% sulphur dioxide, is cleaned of dust by Cottrell electrostatic precipitation and water washing. The gases go straight to five standard contact sulphuric acid plants.

Sulphuric acid

When the sulphur dioxide gas concentrations are low, aqua ammonia is used as an absorbent as previously mentioned. The addition of sulphuric acid to the ammonium sulphite solution produced results in the liberation of almost pure sulphur dioxide and the formation of ammonium sulphate The latter is pumped to a separate plant for crystallisation. To make sulphuric acid from the regenerated gas, the pure sulphur dioxide and by-product oxygen are added continuously to a circulating load of inert gas, and passed through a converter where the dioxide unites with oxygen to form sulphur trioxide in the presence of a vanadium catalyst. The



The rock plant (*foreground*) and storage plant (*background*) are two of the main buildings at Kimberley. The storage plant has a capacity for about six months' production. In the rock plant, phosphate rock is subjected to coarse crushing and then stockpiled. Just before use it is given fine grinding treatment and pumped to the phosphate plant.

gas is absorbed and combined with water to make sulphuric acid.

The standard contact process for sulphuric acid manufacture is used in the five other plants. Over a vanadium oxide catalyst, the purified sulphur dioxide gas combines with oxygen to form sulphur trioxide. The latter is then absorbed in 98.5% sulphuric acid. The final product is largely 93% acid and is obtained by the dilution of the absorption acid.

The total capacity of the six plants is 1,200 tons of sulphuric acid daily. One of the plants is equipped so that it can make oleum of any required strength.

Since the production of sulphuric acid is dependent on the sulphur dioxide gas piped from the metallurgical departments the two plants are situated near to each other. The sulphuric acid manufactured is pumped one mile through a pipeline system and raised 400 ft. to a plateau, on which the other chemical plants are placed. Steam, ammonia, hydrogen, nitrogen and other materials are also transported by pipeline.

Sulphate of ammonia

Although the sulphate of ammonia plant is designed to operate either by direct neutralisation of the sulphuric acid with ammonia, or by evaporating and crystallising the sulphate of ammonia solution formed, the former is seldom used now because all the ammonia in excess of requirements for ammonium phosphate and *Nitraprills* is used in sulphur dioxide recovery.

With a capacity of 600 tons a day the sulphate of ammonia plant is equipped with Oslo evaporators and crystallisers, centrifuges and driers, in addition to standard conveying equipment. The sulphate of ammonia liquor is evaporated and crystallised, after which the crystals are centrifuged and dried.

Ammonium phosphate

Phosphate rock for the ammonium phosphate fertiliser is mined by a Cominco subsidiary in Montana, U.S.A. For the production of concentrated fertilisers the rock must first be exposed to the action of sulphuric acid so as to form phosphoric acid. This is accomplished at Trail in two units having a total capacity of 800 tons of phosphate rock a day. The reaction takes place in large mechanically agitated tanks. The mixture is then filtered. This separates the phosphoric acid from the gypsum by-product. The acid produced contains 32 to 33% phosphorus pentoxide (P_2O_5).

For the production of fertiliser from the acid so produced there are three units at Trail with a total capacity of 900 tons a day. The slurry formed by mixing phosphoric acid with ammonia gas is ammonium phosphate. To convert this into a medium that can be handled more conveniently, fines produced in process are mixed with the wet slurry in the proportion of about 1:1. These are stirred in a blunger to form a dough-like mixture of fertiliser, which, on drying, forms into pellets. After screening the product is sent to storage.

Two kinds of ammonium phosphate are produced–ammonium phosphate 11-48-0 produced by partial neutralisation of phosphoric acid with ammonia gas, and ammonium phosphate-sulphate 16-20-0 produced by partial neutralisation of a phosphoric acid-ammonium sulphate solution with ammonia gas.

Ammonium nitrate

Nitric acid for the fertiliser ammonium nitrate or *Nitraprills* is manufactured by oxidation of ammonia and subsequent water absorption of the nitrogen oxides produced. The ammonia is burnt in air by passing it over a red hot platinium gauze catalyst. The nitric acid is neutralised by ammonia to yield an ammonium nitrate solution, which is concentrated by evaporation and 'prilled.'

At the Turner Siding, Calgary, chemical plant of Cominco, ammonia and *Nitraprills* are produced. Natural gas is the main raw material. Made up chiefly of methane, this gas is burned in air to produce nitrogen in one section of the plant. Elsewhere, it is reacted with steam to produce hydrogen. The two resultant gases are then purified and converted into ammonia, some of which is used at Calgary, the remainder being transported to Trail. The manufacture of ammonium nitrate at Calgary follows a similar pattern to the process at Trail.

The new $9 million plant now being finished at Kimberley will produce 11-48-0 ammonium phosphate. The suspension roasting of iron sulphide tailings from the Sullivan concentrator will produce about 270 tons of sulphuric acid daily. This, together with ammonia shipped from Cominco's Calgary plant and phosphate rock from the company's mines at Montana, U.S.A. will be the chief raw material.

For research and development Cominco have up-to-date laboratories and pilot plants. There is a modern Central Research Laboratory completed in 1942 and branch laboratories in the zinc department in the Chemicals and Fertilisers Division at the Trail and Calgary plants and at the Sullivan concentrator, Chapman Camp, B.C. In addition, members of the research staff are at work testing and operating pilot plants, etc., in the various departments.

Bearing the trade name 'Elephant Brand,' Cominco fertilisers are shipped to many parts of the world.

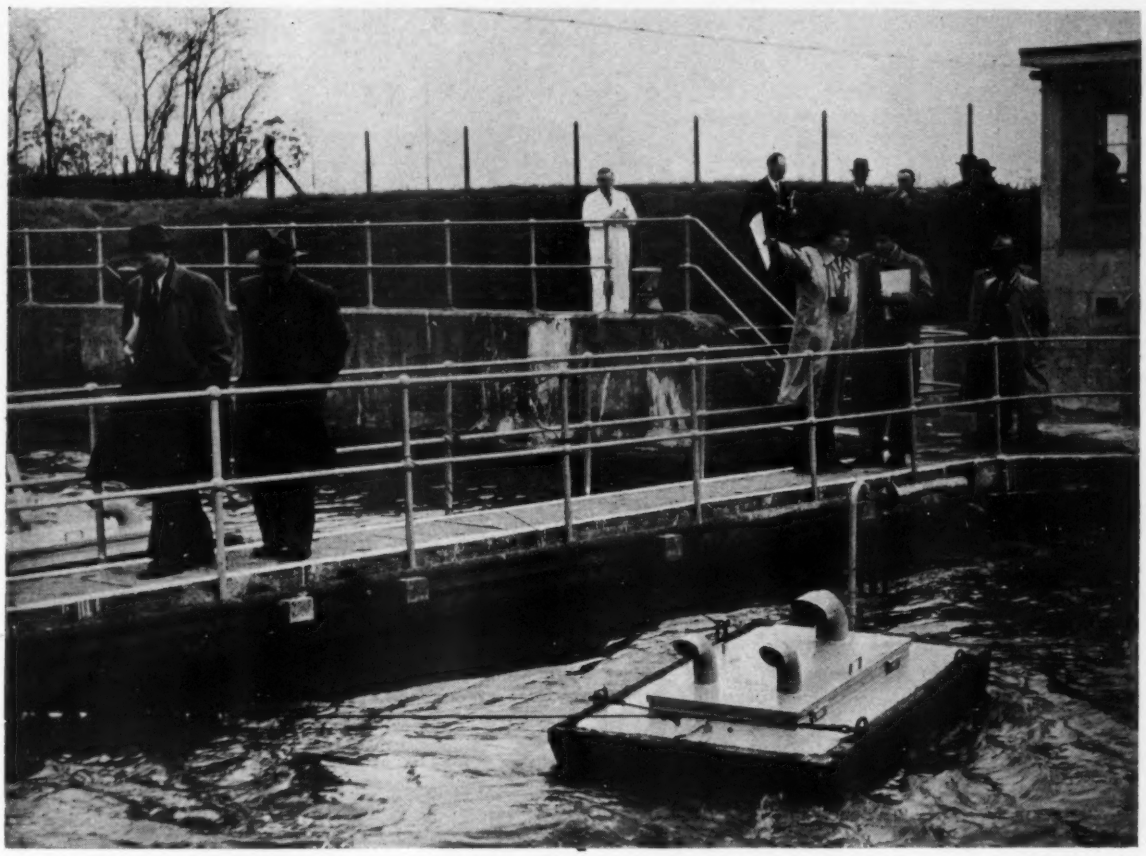
Contributions and Correspondence

THE EDITOR *welcomes practical articles and notes on chemical engineering and industrial chemical subjects with a view to publication. All contributions, which should be fully illustrated whenever possible, are carefully considered. A preliminary letter or synopsis is advisable. Address such material to* THE EDITOR, CHEMICAL & PROCESS ENGINEERING, Stratford House, 9 Eden Street, London, N.W.1.

Letters for publication on any of the subjects covered by this Journal are also invited.

Effluent Treatment at a Fine Chemical Factory

At the Dagenham (Essex) factory of May & Baker Ltd. a wide variety of inorganic and organic fine chemicals and pharmaceuticals is produced which, in the course of manufacture, gives rise to an almost equal variety of waste products for disposal. This variety of effluent problems was, no doubt, the reason why the factory was visited by the O.E.E.C. Mission for the study of water and air pollution problems of the European chemical industries which recently toured England. Here is a description of the effluent treatment and fume absorption system in use at Dagenham. Of particular interest are the 'motor boats' equipped with stirrers for agitating the contents of the shallow treatment tanks.



The O.E.E.C. team inspecting effluent treatment tanks at Dagenham. One of the 'motor boats' used for agitating the effluent in the large shallow tanks is seen in the foreground.

PRELIMINARY treatment, before the effluent leaves the factory, is designed to 'smooth out' fluctuations as far as possible and to bring the *p*H of the final effluent to a figure of not less than 5 and preferably within the range of 6 to 9. This question of acidity is particularly important, since the main sewer from the factory outfall to the local sewage disposal works, where the effluent is finally treated before discharge to the river, is constructed in concrete.

To this end, the factory effluent, including all domestic sewage, etc., is conducted *via* a 24-in. earthenware 'acid drain' to the treatment plant illustrated in the photograph. This consists essentially of two concrete tanks lined with bitumen and each holding approximately 50,000 gal. The relatively shallow depth of these tanks (3 ft. 6 in.) was necessitated very largely by the levels of the existing inlet and outlet sewers, and the desire to avoid pumping the effluent either into or out of the treatment plant.

With both tanks in operation, an average holding period of approximately 2 hr. is achieved—sufficient to ensure reasonable uniformity in the final effluent, and to obtain a substantial degree of self-neutralisation.

On balance, however, the amount of acid effluent discharged from the various processes is greater than that of alkaline, and it is necessary, therefore, to arrange for the addition of alkali in the treatment plant. Because of this overall acidity and since the discharge during short periods of effluent of *p*H above 10 is not harmful, no arrangements are made for the addition of acid.

Neutralisation of the excess acid is

carried out by the controlled addition of a mixture of lime ($Ca(OH)_2$) and chalk ($CaCO_3$) in the form of a slurry. The lime rapidly neutralises the bulk of the free acid, while an excess of chalk acts as a 'buffer' against the possible development of acidity after the effluent leaves the treatment tank but without causing an excessively high *p*H.

The lime/chalk slurry is prepared in a 3,500-gal. agitated concrete tank, from whence it is circulated at approximately 100 gal./min. through a 3-in. ring-main. From this ring-main a single, remote-controlled off-take feeds either or both tanks at a point adjacent to the raw effluent inlet. The rapid circulation through the ring-main prevents settling of the slurry and choking of the pipeline during periods when little or no flow into the treatments tanks is necessary.

The rate of addition of lime slurry is controlled manually. A simple *p*H indicator is installed in the inlet sewer to give warning to the operator of the arrival of excessively acid or excessively alkaline effluent. He also receives advance warning by telephone when strongly acid residues from certain of the larger plants are about to be discharged. By this means, rough adjustments can be made, in good time, to take care of strongly acid or strongly alkaline 'peaks' before the *p*H of the main contents of the treatment tanks begins to change.

Final control of the treatment is by means of a sensitive *p*H recorder placed in the outlet flume from the tanks. Owing to the large volume of liquor in the tanks, rapid changes in *p*H of the treated effluent are smoothed out and, under normal conditions, only occasional adjustments to the flow of lime are necessary in order to maintain the *p*H within the desired range.

Since the entire treatment plant is operated by one man, the expense of fully automatic *p*H control is not justified. Owing to the somewhat isolated situation of the plant and the disastrous results which could follow pump breakdown, power failure, etc., it would still be necessary, even with automatic control, to have an operator on duty throughout the 24 hr.

As an insurance against power failure, a standby petrol engine is installed to drive the agitator of the lime slurry tank. In such an emergency, neutralisation would be carried on by allowing an excess of lime/chalk mixture to overflow from the slurry tank directly into the outlet flume.

Agitator 'boats'

Much consideration was given to the question of the most economic way of stirring the contents of the two 50,000-gal. treatment tanks, which had to be of large area in relation to their depth and which would often contain corrosive liquor. The solution adopted was to moor two small rubber-covered steel 'motor boats' in each tank. These boats are each fitted with a 5-h.p. electric motor driving a standard 18-in. bronze propeller and are moored by means of steel hawsers in positions which have been found by experiment to give maximum turbulence. The advantages of this system are its low initial cost, reliability and extreme ease of maintenance, while its efficiency can be judged from the fact that a quantity of dyestuff placed in the inlet was evenly distributed throughout the 50,000 gal. in 2 min.

Other points of interest are:

No settling of the treated effluent to remove suspended solids is necessary, as a sufficient quantity of waste water is discharged into the 'acid drain' to keep the contents of suspended matter to a reasonably low figure.

The discharge of waste products likely to cause difficulty during subsequent treatment at the sewage farm (*e.g.* sulphides, cyanides, phenol, etc.) is, in general, very small and no special precautions are required. Discharge of certain types of waste is prohibited without special consent. Others are limited to a fixed maximum quantity per day.

Besides the continuous *p*H control, a representative bulk sample of the effluent is taken over each period of 24 hr. and examined for oxygen demand and suspended solids content, the local authorities' treatment charge being based on the findings. This sample is taken automatically by means of a continuous sampling device which delivers approximately 10 c.c. of the treated effluent every 2 to 3 sec. into a bulk container.

Fume absorption

For the absorption of gaseous effluents, the equipment most generally used comprises a Raschig-packed column of appropriate dimensions through which a suitable absorption medium is circulated. If necessary, an exhaust fan is used to draw the vapours through the column. A variant of this system, which is only adopted on the smaller plants, is the use of an ejector in place of the packed column. This has the advantage that it provides the necessary suction automatically and gives very good mixing of the vapour with the absorption liquid. It also occupies considerably less space. A disadvantage is its inability to deal with large fluctuations in the volume of effluent gas.

The following are typical examples of fume absorption which were seen by the O.E.E.C. Mission:

(1) Recovery of hydrochloric acid from a chlorsulphonation process. This is carried out in a standard Kestner unit in which the bulk of the absorption takes place in a film absorber through which weak hydrochloric acid is circulated. In a secondary tower, the residual gas is absorbed in water, and this is fed into the main system as 'make-up.'

In this way, concentrated hydrochloric acid is obtained of satisfactory purity and in sufficient quantity to supply almost the entire requirements of the factory.

(2) General fume disposal from a group of processes, involving phosphorus oxychloride, hydrochloric acid, sulphur dioxide and ammonia.

In this case the exhaust gases are heavily diluted with air and two separate systems are installed —one taking the acid, and the other the alkaline vapours. In each case, two scrubbing towers connected in series are used with circulation of sodium hydroxide in the acid scrubbers and water in the alkaline.

(3) Absorption of nitric oxide (NO) evolved during the solution of bismuth metal in nitric acid.

Oxygen is fed into the reaction to effect oxidation to NO_2. This is partly absorbed in a small quantity of water and returned to the reaction vessel in order to economise on the usage of nitric acid.

Final absorption is carried out in a glass scrubbing tower using 'aerated' water as the absorbent.

(4) Treatment of effluent gases containing bromine in the presence of a large volume of steam.

This is a normal power-circulation system using sodium carbonate solution, except that it is run at boiling point to prevent dilution of the absorbent solution. When exhausted, this solution is returned to the process to recover the sodium bromide.

The general principle is to install a separate absorption unit on every plant where obnoxious vapours are evolved, and the above examples will serve to demonstrate the basic design of the equipment used. The size, materials of construction and the absorbent must, of course, be varied to suit the individual process.

Laboratory glassware. Quickfit & Quartz Ltd. have issued a new catalogue which lists and illustrates typical pieces of apparatus built entirely of standard Quickfit components in general use. Apparatus of a more specialised character, such as *Soxhlet* extractors, is also shown. Where more than two components are involved the assembly is provided with its own catalogue number to simplify ordering.

'Unidirectional' retractable soot-blowers is the title of a recent Babcock & Wilcox pamphlet. It describes a new type of electrically operated sootblower which does not require reversal of the operating motor, thus simplifying automatic sequence control of sootblowing in large boilers. Photographs and diagrams show the construction and operation of this device.

Plant and Equipment

Measuring oxygen and hydrogen in gases

A recently developed instrument for the continuous measurement of small quantities of oxygen or hydrogen in mixtures of these two gases, or separately in other 'inert' gases, is based on the measurement of the heat of combination of oxygen and hydrogen. The instrument is known as the *Deoxo* indicator and is made by Baker Platinum Ltd. It employs a precious-metal catalyst similar to that used in the *Deoxo* purifiers and, therefore, it is necessary when measuring oxygen to have at least a slight excess of hydrogen (or of oxygen if hydrogen is being measured).

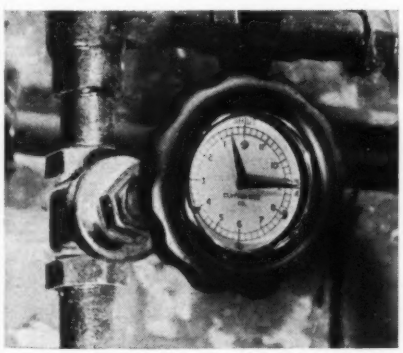
The catalyst is contained in a specially constructed vacuum-jacketed calorimeter, and a multi-junction thermocouple measures the difference in temperature between the incoming and outgoing gas. Thus an e.m.f. is produced which enables continuous indication and, if required, a record to be obtained. Although all the gas being measured is combined on the catalyst, the indicated temperature rise is significantly below the theoretical figure. This is accounted for by heat losses, but, by controlling the flow at a predetermined figure, reliable reproducible results are obtained.

To calibrate the instrument the inlet gas is first passed through a *Deoxo* purifier (built in the instrument for the purpose). Known quantities of oxygen and or hydrogen are then added by means of a small electrolytic cell, before passing to the calorimeter. The electrical output of the instrument is directly proportional to the oxygen or hydrogen content of the gas, *i.e.* the calibration curve is linear and, therefore, calibration at one point only is required. The temperature yield, *i.e.* the indicated temperature rise compared with the theoretical, is of the order of 65%, but the actual figure is not important so long as it remains substantially constant.

For calibration it is, of course, necessary to control the gas flow rate accurately, but, in use, a variation of up to $\pm 10\%$ does not affect the reading significantly. A sensitivity adjustment enables the indicator reading to correspond directly with the oxygen or hydrogen content. The instrument is claimed to cover the range 0.01% up to 1% oxygen with good accuracy and can, with decreasing accuracy, be made to indicate down to as low as 0.001%.

The working of the apparatus would be affected by the usual 'poisons' which inactivate the *Deoxo* catalyst and, therefore, the instrument includes scrubbing and drying bottles. Carbon monoxide inhibits the catalyst and, since it cannot readily be removed from the gas stream, the use of the indicator is not recommended when carbon monoxide is present. However, this gas is an inhibitor rather than a poison, in so far as its effect is not permanent, *i.e.* the catalyst soon recovers its full activity on passing CO-free gas.

A new instrument for the detection and continuous measurement of traces of oxygen and hydrogen in gases.



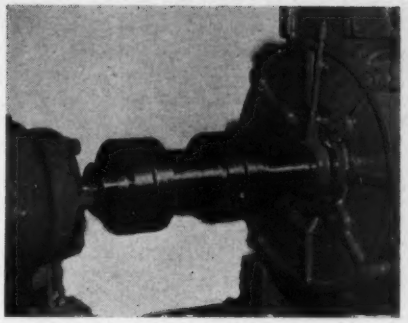
Valve position indicator which can be fitted in place of the handwheel.

Valve position indicators

Not every pipeline can be equipped with a flow meter to indicate the amount of fluid or gas passing through at a particular time. A simple solution of this problem is to fit a valve position indicator, such as was described in CHEMICAL & PROCESS ENGINEERING, 1953, **34**, (1), 26. This can be fitted to any standard valve, with rising or non-rising spindle, whether the valve is in the vertical, horizontal or inclined position. The instrument is fixed in a position co-axial with the handwheel by a bracket attached to the pipe.

A new valve position indicator has now been developed which takes the form of a handwheel incorporating the instrument. Thus no supporting bracket to clamp under the gland nut or around the pipe is required, nor any other obstruction which might interfere with valve manipulation. It is only necessary to remove the standard handwheel of the valve and put the position indicator in its place.

Rotation of the handwheel is transmitted through a small gear train to the indicator hands and the amount of movement is shown on the dial, which is counterbalanced to prevent its turning. The dial is calibrated to one-forty-eighth of a complete turn. Makers of the instrument are Clayton Engineering Co.



A 'spacer' type coupling, which allows a large degree of angular and lateral misalignment, fitted to a Pulsometer Pacific process pump.

Flexible metallic couplings

The need for reasonably free axial movement between the driving and driven halves of flexible couplings for pump and compressor drives has led to the development, by Metaducts Ltd., of a coupling which allows for misalignment and needs no lubrication. In the *Metastream* coupling, as it is called, transmission is through flexible discs or diaphragms, so that the only resistance to relative axial movement between driving and driven halves is that imposed by the load/deflection characteristics of the membranes themselves. Three types of coupling are made:

Non-spacer M type. This was developed primarily for oil refinery and power station services. The coupling employs two banks of membranes, assembled in an integral membrane unit which can be removed complete without disturbing either of the driving or driven half machines or half couplings.

Spacer M type. In its essentials, this comprises two *Metastream* couplings with a connecting spacer or distance piece. The spacer permits the coupling to take a large degree of angular and lateral misalignment. The spacer and membranes can be removed individually, or complete as a sub-assembly unit, without disturbing either the driving or driven half couplings or machines. In oil refinery practice the gap between driving and driven half couplings, when the spacer unit has been removed, permits removal of the pump or other driven machine rotating element.

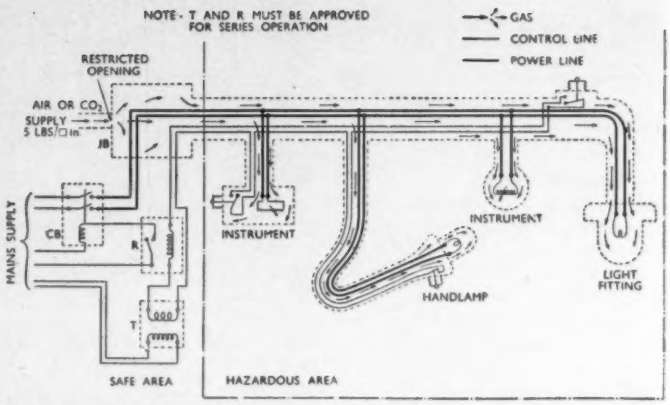
Industrial I.M. type. This incorporates the *Metastream* features, but can be used for simple industrial drives. Here again the membrane unit is removable.

Lighting system for hazardous atmospheres

The design of electric lighting equipment for hazardous areas is a difficult problem, particularly where the gases classified in Group IV in British Standard 229 are present. These comprise hydrogen, acetylene, carbon disulphide, blue water gas and ethyl nitrate. Even in other gases, portable lighting apparatus is often essential and, while the fitting can be made flameproof, the connecting cable, however well protected, is never infallible.

One way in which the difficulty has been overcome is by encasing the wires and apparatus in an envelope of air or other non-inflammable gas such as CO_2. Any fracture of the containing tube or apparatus results in the escape of the compressed air with a reduction in pressure. This operates a pressure valve situated near the main switch which isolates the building or section from the supply. A drawback is the time lag before the current is cut off, owing to the draining of the whole system of air, and also, in the case of a portable and compressible tube (which must be used for a portable lamp), the likelihood of the tube being compressed by a weight on it and so throttling the air supply and isolating the control switch.

A new pressurised system, developed by the General Electric Co. Ltd., is claimed to overcome these disadvantages. The whole system depends on the operation of the special pressure-responsive switch, which is a micro switch held in contact against a spring bias and kept closed by a positive air pressure within the system. The control system is fed from a transformer through a relay. Both the transformer and relay are certified as being intrinsically safe for use in the appropriate gases, including hydrogen. The circuit is made through all the pressure-responsive switches in the system, or an appropriate part of the system, connected in series. Thus, should any switch open, the current is instantly cut off by the control relay tripping the circuit breaker. This happens when a fracture occurs in a casting or cover glass of a lamp, a break in a flexible tube or conduit, a leakage in a switch, relay or fuse cover, or failure of the air supply. Another protective feature is that, when such fracture or leakage occurs, the enclosed air emerging through the fracture or leak keeps away the surrounding gas-laden atmosphere for the very limited time which elapses until the current is cut off.



Schematic diagram of a new pressurised system for electrical control in hazardous atmospheres. Key to components on left: JB—barrier coupling box, CB—circuit breaker, R—relay, T—intrinsically safe transformer.

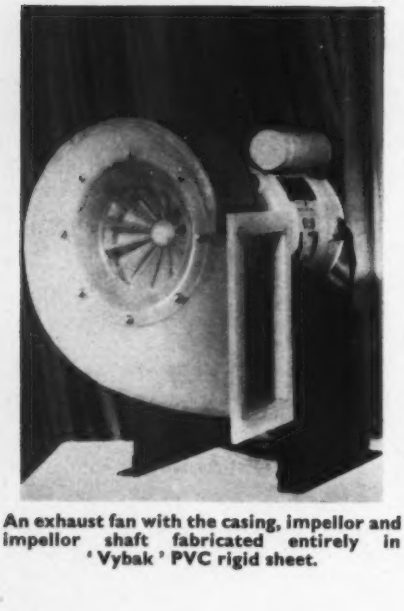
The system can be used for controlling a portable lamp. Where fluorescent lighting is required, a special fitting is available incorporating a 2-ft. tube in a *Perspex* cover.

Small continuous fat-splitting plant

A small plant for continuous fat splitting has been designed by the Blaw-Knox Co. It utilises the Colgate-Emery process and has a capacity of 500 lb./hr. The plant is claimed to provide a high degree of split, up to 98%, coupled with low operating costs. Larger plants with capacities ranging from 1,500 to 7,500 lb./hr. have been operating successfully since 1947 producing high-quality fatty acids and glycerin. The fat-splitting column, 65-ft. high, is unhoused and requires a ground space of only 5-ft. square. The accessories require a building space 10 × 15 ft. and 16 ft. high.

For nearly a century fat splitting had been a batch operation, and for some 50 years a catalyst had been used to speed up the hydrolysis. The Colgate-Emery process makes it possible to eliminate the catalyst and has the advantage of being a continuous, counter-current reaction.

Briefly, the process consists of continuously passing fat and water in counter-current flow in a pressure column, in the central zone of which the water, in small drops, falls slowly through the fat at a temperature of 500°F. The materials are introduced at normal pumping temperatures, and are heated in the column principally by direct heat contact exchange with the product. The balance of heat required is supplied by injecting direct high-pressure steam. The products, fatty acid and sweet water, are relatively cold when discharged from the column. The column is completely filled with liquids and boiling is prevented by maintaining a pressure in excess of the boiling point of water. Because of the direct heat exchange, less steam is required than in other comparable processes.



An exhaust fan with the casing, impellor and impellor shaft fabricated entirely in 'Vybak' PVC rigid sheet.

New exhaust equipment

A new series of exhaust fans, coupled with a new form of ducting, have been developed by Turner & Brown Ltd., Bolton. Known as the *Turbo* range of centrifugal fans, they and the ducting are almost entirely constructed of *Vybak* rigid plastic industrial sheet, which is supplied by Bakelite Ltd. and which was described in our November 1953 issue.

The fans range from the Junior model, with an extraction capacity of 300 to 330 cu.ft./min. and intended for use with fume cupboards, small laboratories, etc., though a variety of sizes to suit individual requirements up to a maximum displacement of 15,000 cu.ft./min. They are driven by fractional-h.p. motors at a speed of 1,450 r.p.m. in the case of the Junior.

In these fans the casing, impellor and impellor shaft are made entirely from *Vybak*, and the whole is so designed that no metallic parts are in contact with corrosive fumes. The casing itself conforms to existing principles, but is bolted to an outside frame of metal, and this arrangement allows the outlet to have 16 equi-arc positions as well as permitting the fan to be easily coupled with existing ducting or fitted into small, enclosed areas. The impellor has 12 blades round a central boss, all made from the same material, and a hollow shaft to allow the introduction of the keyed steel shaft from the motor. The price of the fans, complete with motor, is £41 each ex works.

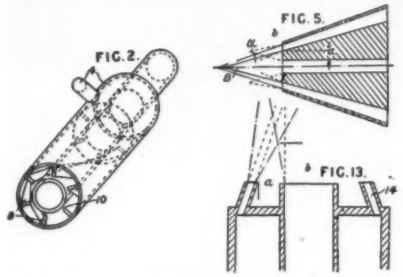
For further particulars of plant and equipment please use the coupon on page 70.

Chemical Engineering Invention

MONTHLY SUMMARY OF PATENT CLAIMS

Production of metal and silicon oxides

Silicon, titanium and zirconium oxides are produced by decomposing the chlorides with oxygen-containing or oxygeniferous gases at high temperature, the gases passing to the reaction chamber in concentric flow and one at least being in a state of spin. When both are spinning, the spin may be in the same or opposite direction. An inert gas, *e.g.* nitrogen, carbon dioxide or chlorine, or one which is inert only towards the chloride, *e.g.* hydrocarbons, carbon monoxide, hydrogen or mixtures thereof, may be used to separate the reactant gas streams, or it may be fed in around the streams. Examples indicate a temperature



of the various streams varying from 100 to 900°C.

A variety of nozzles is shown. In Fig. 2, the outer feed duct 4 is subdivided by helical partition walls 8, the edges of which may be spaced from the inner duct wall at 10. The helical walls need not extend to the outlet aperture. Fig. 5 shows a nozzle of truncated cone structure, the helical walls not being shown. By varying the angles α and β and the distance a, the mixing speed and distance b from the nozzles at which the gas current contact may be varied. Another form is shown wherein helical walls are disposed in the inner duct, and a further form with said walls in both ducts. There is also shown a core member formed with helical flutes which may be pushed into the central delivery tube. Three further forms are illustrated, with three concentric ducts, helical walls being disposed respectively in the outer, outer and inner, and all three ducts. In the last, the intermediate helical walls are opposed in direction to the outer and inner helical walls.

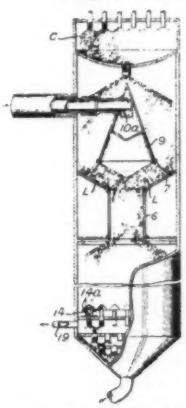
In Fig. 13 the outer duct is subdivided into straight channels 14 arranged in skew position with respect to the central axis of the nozzle. The channels may be arranged in a circle, ellipse or on any other figure around the central duct. Dimensions corresponding to a and b of Fig. 5 may be varied. Other arrangements include one or more circles of channels in combination with plain or helically-divided ducts. Channels 14 may be formed as bores in a thick plate. Specifications 535,214 and 567,093 are referred to.

The Specification as opened to inspection under Section 91 refers to the production of metal oxides generally, the decomposition of ferric chloride and aluminium chloride being referred to. This subject matter does not appear in the Specification as accepted.—655,647, *Saurefabrik Schweizerhall.*

Houdry cracking process

In cracking or viscosity-breaking hydrocarbons in the presence of granular contact material, the latter is fed downwardly through a conversion zone as a compact non-turbulent bed. Liquid hydrocarbon is atomised in a space within the bed and directed on to the surface of a layer of material which flows freely beneath and forms the lower boundary of the space. The cracking or viscosity breaking takes place during subsequent passage of the layer of contact material to a bed of material in the lower part of the chamber. The hydrocarbon need not be fully vaporisable; the contact material may be catalytic when cracking material to produce gasoline and fuel oils at a reaction temperature of 425 to 540°C., or may be substantially inert, *e.g.* fused alumina quartz, fused silica and alumina when treating viscosity-broken distillates at 425 to 675°C.

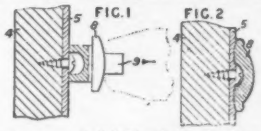
As shown, contact material C is fed above a conical deflector 9 which builds it up into a layer formation *L* on a dish-like member 7. The layer slides down the



dish out through tube 6 into the lower part of the chamber where, in passage through tubes 14, the treated hydrocarbon vapours are removed through passages 14*a* and taken out through pipe 19. The heated hydrocarbon is injected through nozzle 10*a* into a space within the downwardly moving bed formed by conical deflector 9 and on to the layer *L* of contact material.—647,591, *Houdry Process Corporation.*

Thermoplastic lining of chemical plant

In the lining of chemical apparatus with sheets 5 of thermoplastic material such as polyvinyl chloride the fixing screws are covered by caps of the same or other thermoplastic material welded in place. The cap has an enlarged body part 8 with an annular flange to cover the screw head and a square projection 9 for gripping in the chuck of a portable drill. The cap is pressed on to the sheet whilst rapidly rotating and is friction-welded in place as indicated in Fig. 2. The part 9 may be sawn or filed off.



The caps may initially be of the general shape shown in Fig. 2 and be welded on by heat.—645,114, *Communications Patents Ltd., G. Haim and H. P. Zade.*

Dyes reduce fertiliser caking

The tendency to caking or setting of ammonium sulphate crystals is reduced by distributing over the surfaces of the crystals a deposit of a dyestuff of sulphonated aromatic character. When the dyestuff is dissolved in a hot saturated aqueous solution of ammonium sulphate and the resulting solution is cooled to incipient crystallisation, the ammonium sulphate crystallises in the form of thin fragile crystals. The dyestuff, *e.g.* in amount of 0.001 to 0.1 parts per 100 parts of ammonium sulphate, may be added to a saturated aqueous solution of ammonium sulphate and the latter crystallised by cooling. Alternatively, ground ammonium sulphate may be mixed with water containing the dyestuff and the water evaporated from the uniformly dye-covered crystals. Again, ammonium sulphate crystals, the form of which may be modified by crystallising in the presence of aluminium sulphate, may, in their wet state from crystallisation, be sprayed with a solution of the dyestuff and dried.

Dyestuffs specified are: Naphthol Green BNS, Fast Yellow, Naphthalene Fast Orange, Azofuchsine G, Ponceau 6R, Chromazol Yellow, Chlorazol Skye Blue, Fast light Yellow, Lissamine Fast Yellow, Disulphine Blue V, Disulphine Blue AS, Xylene Cyanol FF, Brilliant Cochineal, Bordeaux, Crystal Ponceau, Carmoisine WS and LS, Naphthalene Red EAS, Bismarck Brown, Hydrazine Yellow SO, Eriochrome Red, Acid Green G, M and GG Extra, Erioglaucine Extra, Fast Acid Violet 10B, Acid Violet 4BNS, Edicol Amaranth and Tartrazine, the last two being particularly suitable.—649,240, *A. Butchart, J. Whetstone and Imperial Chemical Industries Ltd.*

Vinyl Resin Manufacture

BAKELITE'S NEW PLANT AT AYCLIFFE, DURHAM

ALTHOUGH Bakelite Ltd. are best known for their phenolic and urea thermosetting products—moulding materials, laminated sheet and resins—the company have, since the early days of the last war, been active in the thermoplastics field. It was in 1943 that they started the manufacture of vinyl resin compounds for cable covering at their dispersal plant at Feniscowles, Lancashire.

These compounds were urgently required to replace the rubber lost when the rubber-producing countries fell to Japan. Primarily produced for electric cable insulation and for petrol tubing, the uses of vinyl resins and their fabricated products have steadily increased in number. These demands have continued to grow in spite of the fact that rubber has again been freely available for years, because vinyl resins have many important properties not enjoyed by rubber.

At the end of the war the company closed down their several dispersal factories, returning to the main works at Tyseley, Birmingham, such plant as could be accommodated there. Expansion at Tyseley was limited by a number of factors and, after consultation with the Board of Trade, it was decided to develop a site at Aycliffe, Co. Durham, primarily for the production of thermoplastic products, of which vinyl-type polymers are a very important group.

A 35-acre site was selected which included a number of Royal Ordnance Factory buildings. Some were speedily adapted to take the vinyl compounding equipment from Feniscowles, and others used for administration, laboratories, stores, engineering and other services. However, these new facilities were insufficient and a building programme was immediately put in hand. The first phase was completed in 1948. The new buildings house a unit for the manufacture of phenolic moulding materials which is probably the largest and most up-to-date in the world, plant for the production of urea-moulding materials and for compounding, calendering and press polishing PVC.

The second phase of the building programme was a new resin plant, and this plant has now come into operation. It occupies part of a 47-acre site which the company has purchased immediately adjoining the original 35-acre site. It is a polymer plant, and consists essentially of low-temperature storage and mixing tanks for monomers, polymerising equipment, drying, grinding and screening plant and weighing and storage facilities to handle the final product.

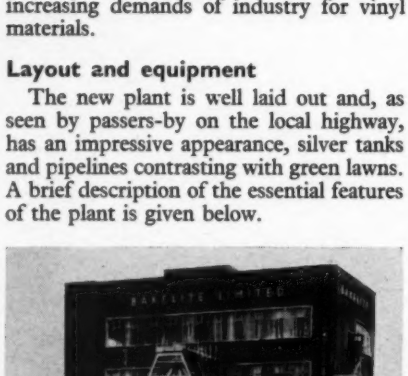
This new plant is designed to produce different types of polyvinyl and copolymer resins. Initial production is on the vinyl

Calendering 'Vybak' PVC sheet.

chloride-acetate copolymer of the *Vinylite* 'VYNW' type, which will be sold under the company's trade mark as *Vybak* 'VYNW.' This is claimed to be an excellent general-purpose resin, giving easier working, fluxing and plasticisation than straight PVC resins without sacrifice of mechanical and electrical properties. Present output is at the rate of thousands of tons per annum and, in addition to supplying the company's own requirements, will be sold to other compounders and so go some way towards meeting increasing demands of industry for vinyl materials.

Layout and equipment

The new plant is well laid out and, as seen by passers-by on the local highway, has an impressive appearance, silver tanks and pipelines contrasting with green lawns. A brief description of the essential features of the plant is given below.



The vinyl resin plant from the south west, with the storage and weighing bins in the foreground.

The services, which are integral with the plant, comprise circulating water equipment and water storage, fire-fighting equipment, refrigeration and nitrogen units. The largest user of cooling water is the refrigeration plant. Nitrogen is required as an inert blanket in all pieces of equipment in which vinyl chloride is either stored or polymerised, and to keep free of vinyl chloride, instruments such as pressure gauges and containers where blockage by the polymer would result if the monomer was not excluded. Refrigeration is supplied to the reaction and storage areas as calcium chloride brine, which is produced by an ammonia compression refrigeration plant.

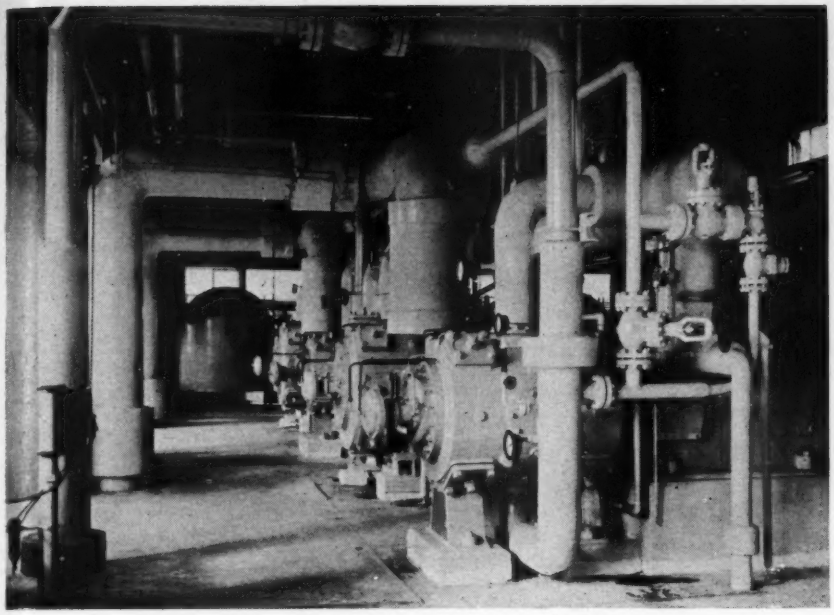
While normal operation of essential services and operating equipment depends on electric motors, steam-driven stand-by plant, as well as diesel-driven generator equipment, automatically take over in the event of failure of the mains power supply.

Raw materials arrive on site in road tankers and are off-loaded by pumps or gas pressure into the storage tanks which are grouped together in a tank area. From the storage tanks they are pumped to weigh tanks to make up feed mixtures of varying compositions corresponding to the type of resin being made, and these fed to the polymerisation system as required. The polymerisation system can manufacture resin by any of the well-known techniques, and is designed to give the flexibility necessary to cover a wide range of copolymer products with good material and service efficiencies. A remarkable feature of all this section of the equipment is that, for the first time in this country, it is out of doors, though the final drying of the resin, its grinding, screening, blending and packing are carried out indoors.

The equipment is entirely British made, and extensive use is made of stainless steel to ensure a clean product and freedom from corrosion.

Processing

Manufacture is controlled at all stages by the most modern servo-control mechanisms available, and extensive use of these has enabled the plant to be operated with a small labour force. Control of raw materials and intermediate products is covered by a qualified technical staff, covering the 24-hr. service on a seven-day week basis necessary to ensure full cover during all periods of manufacture. Special attention is given to ensure that the finished resin is uniform in all characteristics, and it is tested not only for its physical and chemical properties as a resin, but also by its behaviour when used in a variety of compound formulations specially designed to show up any defects. The laboratories



A view of the refrigeration plant showing the compressors.

devoted to the aspect of manufacturing control are fully equipped with the chemical and physical equipment necessary to do the complete evaluation considered necessary by the company.

The resins produced in this new polymer plant are either converted on the site into moulding or extrusion compounds or sheet, or despatched to customers who do their own conversion.

Initial compounding for both moulding and sheet materials follows the same course: through ribbon-type pre-blenders, Banbury mixers and two-roll mills. For moulding and extrusion compounds, strips are fed into granulators where they are cut into chips of various mesh to suit different applications.

For the production of sheet, the Banbury and milled compounds are fed to calenders which produce material up to 54-in. wide and up to 30-thou. thick. The calendered sheet in roll form is then either supplied to customers for further processing—for example, embossing—or is press polished in the company's own presses.

The equipment for compounding, calendering and pressing compares favourably with corresponding equipment in this country and overseas.

Products

It will be realised, therefore, that the thermoplastics products of the Aycliffe plant fall into four groups—copolymer resins, moulding and extrusion compounds, calendered sheeting and rigid and flexible press-polished sheet. They all have good chemical resistance and good electrical properties. They vary from completely rigid to soft rubbery textures, and their colour range is virtually unlimited. Rigid grades are characterised by exceptional dimensional stability. The resins are used in the plant to produce compounds and sheeting and are also sold to customers who prefer to do their own compounding.

The *Vybak* range of extrusion and injection moulding compounds cover all British Standard and Government specifications. They are used, for example, for the coating of electric cables, telephone, radio and car wiring, motor-car-wing piping, garden hose and belts.

The newest of these materials, which are of growing importance, are those used for the production of flexible injection-moulded components which are finding increasing use in various applications in industrial and domestic markets. Rigid extrusions for chemical piping and similar purposes are produced from other compounds and compounds are also supplied for the manufacture of long-playing gramophone records.

Calendered sheeting, which is otherwise used for handbags, wallets and similar purposes, is used as the intermediate form for the manufacture of press-polished sheeting.

These press-polished products are of particular interest, and Bakelite Ltd. has been a pioneer in this country in the production of glass-clear sheets, both flexible and rigid, which are being supplied in increasing quantities to the motor trade. The rigid sheets are used as side screens and the flexible for rear windows in drop-head coupes. For the latter purpose they have the advantage that they do not yellow with age and also that they fold with the hood, thus making possible the use of bigger rear windows and improving the rear view.

Within the last year or two there has been an increasing awareness in this country of the importance of polyvinyl chloride rigid sheet as a material of construction for chemical and allied plant. Techniques for the use of this material have been known and used on the Continent for some time. Bakelite Ltd. produce a rigid sheet suitable for this work. It is inert to most acids and alkalis and can be fabricated using relatively simple equipment. The company is prepared to demonstrate this technique to users.

In addition to the production plant at Aycliffe and the extensive testing facilities, there are well-equipped research and development laboratories, the staff working closely with the works' technical staff. Fundamental research is, however, carried out at the company's research department at Tyseley, Birmingham.

Export Opportunities

Flexible metallic hose

Austen Bros. Ltd., 118 Hollis Street, Halifax, Nova Scotia, want the names and addresses of U.K. manufacturers of flexible metallic hose of all types, including bronze and steel constructions. The hose should be similar to those manufactured by companies such as the American Metal Hose Co., of Waterbury, Conn., and Atlantic Metal Hose Co. Inc., of New York, N.Y., and the fittings and couplings should have the same threads as, and be interchangeable with, American threads.

Catalogues and price lists should be sent to Austen Bros. Ltd. at the above address. (NOTE: British Board of Trade officers in North America report that firms in the U.S.A. and Canada are likely to ignore offers not expressed in dollars and North American weights and measures.)

Sugar factory and distillery plant

The Burmese Ministry of Industry is calling for tenders for the supply, delivery and erection of all machinery, equipment, buildings and auxiliaries for a sugar factory having a cane-crushing capacity of 1,000 long tons per 24-hr. day with 'plantation white' sugar as the end-product by the sulphitation process.

The closing date for the receipt of tenders is February 28. Tenders, in quadruplicate and enclosed in a sealed envelope marked 'Tender for Sugar Factory and Distillery Project' on the outside of the envelope, should be sent to the Secretary, Ministry of Industry, Rangoon. It is understood that those on a 'turn-key' basis after six months' satisfactory operation will be preferred.

For further details of these enquiries, write to the Export Services Branch, Board of Trade, Lacon House, Theobalds Road, London, W.C.1.

New standards

Flow diagram symbols (B.S. 974 : 1953, ' Symbols for use on flow diagrams of chemical and petroleum plant,' price 3s. 6d. net). Since this standard was originally published in 1941 several other standards for graphical symbols in branches of engineering allied to chemical engineering have been issued. Certain discrepancies between corresponding symbols necessitated a revision.

The committee charged with this task, under the authority of the Chemical Industry Standards Committee, proceeded on the principle that, in order to keep the number of basic symbols to a minimum, only those symbols most necessary should be adopted, reduced to their simplest form, and be sufficiently pictorial to enable the units of the plant so represented to be readily recognised. The committee have endeavoured to avoid the use of any symbol which might be identified with a proprietary article. Symbols are intended to indicate the function of a unit of plant, although they may differ in shape from the form of construction of various types of such units all fulfilling the same purpose.

Xyloles (B.S. 458 : 1953, 7s. 6d. net). This revised edition of B.S. 458 (previous edition, 1939) completes the series of revisions of the four British Standards for benzoles and allied products, the other three (B.S. 135 ' Benzoles,' B.S. 479 ' Coal-tar naphthas ' and B.S. 805 ' Toluoles ') having been published in April 1953.

The main features of this revised standard are (*a*) the inclusion of specifications for products of lower specific gravities than those previously standardised, (*b*) the inclusion of two additional grades of xylole of 10° boiling range, (*c*) the inclusion of xyloles of the 3° and 5° boiling ranges which have received a less drastic sulphuric acid treatment than was previously usual, namely, solvent xyloles, and (*d*) the introduction of an improved copper-strip test capable of numerical interpretation, for those products for which it is considered necessary to limit the corrosive sulphur.

Tubular heat exchangers are specified in B.S. 2041 : 1953, which applies to shell and tube heat exchangers for use in the petroleum industry and covers dimensions and manufacturing requirements peculiar to tubular heat exchangers. It should also be read in conjunction with B.S. 1500 (Provisional) ' Fusion - welded pressure vessels for use in the chemical and allied industries ' or any other suitable code which may be specified. The document is divided into various sections dealing with general requirements, materials, construction and workmanship, scantlings, testing, marking and inspection and testing facilities.

Appendices are included, giving details of materials to be used, dimensions of shell, shell cover and channel flanges as well as pressure temperature ratings for tube plates and flanges. Price 12s. 6d. net.

★ PERSONAL PARAGRAPHS ★

★ **Mr. W. Leonard Hill,** chairman of Leonard Hill Ltd., publishers of CHEMICAL & PROCESS ENGINEERING and the 11 other journals comprising the Leonard Hill Technical Group, is undertaking a tour of the West Indies and the United States. He arrives in New York on February 8 and from there goes to Nassau of the Bahamas on the 16th. He leaves Nassau on the 22nd and will arrive in New Orleans on the 23rd. He will then go to Houston, Texas, arriving on the 24th. Then, by way of Dallas and Tulsa, he will go to Knoxville, Tennessee, arriving on March 2. He will return to New York on March 6 and stay there for a month, returning to England on April 1.

While in New York, Mr. Hill can be reached c/o Norris Evans, Esq., 92 Lorraine Avenue, Upper Montclair, New Jersey.

★ **Mr. W. A. Shaw,** A.I.E.E., has been appointed sales director and elected to the board of Evershed & Vignoles Ltd. Mr. Shaw, who succeeded Mr. F. C. Knowles to the position of general sales manager in 1949, studied electrical engineering at the Borough Polytechnic Institute under the late Dr. Henderson. In 1913 he joined the test room staff of Evershed and was for several years engaged in development and experimental work on a wide range of instruments. He transferred to the commercial side in 1919 as a technical assistant and in this sphere he has had a very wide experience in the application of the various Evershed products.

★ **Mr. H. W. Griffiths,** A.M.I.E.E., M.S.M.A., M.I.Ex., has been appointed general sales manager of Evershed & Vignoles Ltd. in succession to Mr. Shaw. He was educated at Battersea Polytechnic and received his early electrical training with the Westminster Electric Supply Corporation and Central London Electricity Ltd. He became the export sales engineer with Evershed & Vignoles. He was appointed export sales manager in 1948.

★ **Mr. L. W. H. Rea,** B.Sc.(Eng.) has been elected to the board of Edwin Danks & Co. (Oldbury) Ltd., following the resignation of Captain (E.) Gregson, R.N.R. Mr. Rea joined the parent company, Babcock & Wilcox Ltd., in 1946 for a four-year student apprentice course. After a period in the Service Department, he joined Edwin Danks and was appointed personal assistant to the managing director. He was appointed general manager of the company in October last.

★ **Mr. W. Smith,** works manager of Edwin Danks, has also been elected a director in place of Mr. J. W. Hargreaves, who has resigned. He was apprenticed to the company in 1910 and obtained a very wide experience of the practical side of boiler making. He was appointed works superintendent in 1940 and works manager in 1946.

★ **Mr. H. S. (" Dick ") Broom,** M.B.E., B.Sc., M.I.Mech.E., has been elected president for 1953-54 of the British Engineers' Association. He had already for some years represented the British Compressed Air Society and the Federation of Manufacturers of Contractors' Plant on the Association Council when he was elected a vice-president in 1951. Mr. Broom is the only son of Broom & Wade's founder and chairman, Harry Skeet Broom, and served his apprenticeship with the company. In 1949 he became joint managing director with his father and his cousin, Mr. C. Broom Smith, and added to his other responsibilities the direction of the sales policy of the company.

★ **Mr. Vaughan Pendred,** sales director of Head, Wrightson & Co. Ltd., of Thornaby-on-Tees, left the U.K. early in January for an extended tour of India. This is being undertaken with **Mr. M. H. Jenks** from the Calcutta office of Balmer Lawrie & Co. Ltd., whom Head Wrightson & Co. Ltd. have just appointed as their selling agents in India.

★ **Mr. M. E. O'Keeffe Trowbridge,** B.Sc., A.C.G.I., A.M.I.Chem.E., has recently joined the head office staff of Sharples Centrifuges Ltd. He was formerly head of the Projects Initiation Department of Head Wrightson Processes Ltd. Before that he was with I.C.I. (Billingham Division).

★ **Mr. L. H. Cooper,** chairman of the Mond Nickel Co. Ltd., has been elected a vice-president of the parent company, the International Nickel Co. of Canada Ltd. He joined the Mond Nickel Co. as chief accountant in 1926 and in 1928 was appointed secretary. In 1945 he became a director of Mond Nickel and its subsidiaries and two years later was appointed assistant secretary and assistant treasurer of Inco of Canada. He became chairman of the Mond Nickel Co. in January 1951.

★ **Mr. A. Lloyd,** previously joint manager of the Thermosetting Division of Bakelite Ltd., and works manager at the Tyseley works, has become general works manager. **Mr. G. J. Taylor,** previously joint manager and sales manager of the Thermosetting Division, is now manager of the Division. **Mr. P. Smith** has become sales manager of the Thermosetting Division; his former assistant, **Mr. C. E. Tofts,** takes his place as sales manager, laminated materials. **Mr. R. Hulse,** previously manager of moulding material production at Tyseley, becomes works manager, Tyseley. His successor is **Mr. A. Tonks,** previously of the works technical department.

World News

GREAT BRITAIN

Titanium oxide plant extensions

British Titan Products Co. Ltd., which, together with its Australian subsidiary, claims to make more than 80% of the titanium oxide manufactured in the British Commonwealth, intends to spend £4½ million on further extending its manufacturing capacity. The demand for titanium pigments continues to outstrip supply, so that the market is still short, although this company alone has spent over £5 million since the war on plant extensions in the United Kingdom, Australia and a share in the Indian factory.

The first extension to the Grimsby plant, at present under construction, will cost £1½ million and will become effective at the beginning of 1955. The second extension, now being planned and involving a major addition to the sulphuric acid plant, will be completed by about the middle of 1956.

When the extensions are completed in 1956 the post-war capital expenditure will be approaching £10 million. An expenditure of £4¾ million is planned for the development of ilmenite property abroad (titanium oxide is manufactured from ilmenite ore) and still further extensions to the Australian plant.

The company's flash roasting pyrites acid plant was fully described and illustrated in CHEMICAL & PROCESS ENGINEERING, February 1953.

Monsanto's new plastics enterprise

A new and wholly owned subsidiary company of Monsanto Chemicals Ltd., called Monsanto Plastics Ltd., has been formed with an initial capital of £100. The purpose of the new company is to consolidate and extend Monsanto's activities in the plastics field. The directors are: J. W. Barrett, D. R. Mackie (commercial director of Monsanto Chemicals), N. F. Patterson, E. L. Pixton, P. A. Singleton (managing director of Monsanto Chemicals) and J. W. Urban. The secretary is R. E. H. Sheridan, assistant secretary of Monsanto Chemicals.

Gilbert Dodd, who is named as general manager of the new subsidiary company, is well known in the plastics industry both at home and abroad. He joined Monsanto's sales division in 1939. In 1949 he was appointed manager of the plastics department, and in 1953 became general manager of Monsanto (Soil Conditioners) Ltd.

Phthalic anhydride shortage

The President of the Board of Trade was asked recently what was the reason for the shortage of phthalic anhydride, which was causing difficulties to synthetic resin manufacturers, and if he would relieve this product of key-industry duty during periods of shortage.

Mr. Heathcoat Amory replied that growing demand for this material and large-scale American purchases in Europe had caused a temporary shortage, which should be ended in 1954 by new capacity coming into production. The possibility of temporary exemption, such as was made between 1950 and 1952, was under consideration.

Evershed's marketing Tinsley instruments

Evershed & Vignoles Ltd. have taken over the distribution of the products made and previously marketed by Tinsley (Industrial Instruments) Ltd. In future all correspondence in connection with Tinsley Instruments should be addressed to Evershed & Vignoles Ltd., Acton Lane Works, Chiswick, London, W.4 (Telephone ELGar 6081).

New pipe-making process

By agreement with the United States Pipe & Foundry Co., the special method developed by the American firm for making long cast cylinders and tubes is to be employed by two subsidiaries of Sheepbridge Engineering Ltd. A prominent feature of this new horizontal rotation process is the use of a special refractory spray round the walls of the mould that induces the casting metal to flow evenly throughout its length, thereby eliminating a principal difficulty in normal British practice.

Sheepbridge Steel Castings Ltd. intend to spin cast piping in high alloy steels and nickel base alloys. Long piping in special alloys is difficult to produce by normal methods. Heat-resisting and corrosion-resisting steel alloys will also be spun in long castings by this new process for ultimate use as rollers in heat-treatment furnaces, runout-rolls for steel mills, etc. Experimental 10-ft.-long iron cylinders of 8 in. diameter have already been successfully cast by the new method, known as the *Burlington* process, at the Chesterfield works of Sheepbridge Stokes Ltd.

Polythene-film company starts trading

British Visqueen Ltd., the joint company to manufacture polythene film formed by Imperial Chemical Industries Ltd. and the Visking Corporation of Chicago, Illinois, is commencing trading this month. Its products will include *Visqueen C*, a special film which overcomes the problem of ink adhesion on polythene film.

The new plant and offices being built at Stevenage will not be completed until July or August of 1954. In the meantime, the company's headquarters will be at 94 Tewin Road, Welwyn Garden City, and plant is being installed in temporary premises at Hillhouse in Lancashire.

The formation of the new company was reported in CHEMICAL & PROCESS ENGINEERING, 1953, **34** (8), 261.

Cannon planning new factory

The building of a new factory was foreshadowed at the annual general meeting of Cannon (Holdings) Ltd. Mr. A. F. Oatley, chairman and managing director, stated that expansion had now reached a point where further development of the existing works was both impracticable and uneconomic. In the past the company had produced multitudinous types of products, functioning primarily as general ironfounders. Since the war it had completed a change to a manufacturing plant producing a few specialised products on mass production lines for which the existing factory, comprising a series of separate buildings, was never designed. The necessity to further increase output, cope with keener competition, and envisage expansions into the export market called for a factory planned for the purpose. Priority was being given to this matter.

Reorganisation of Group. The accounts for the year ended June 10, 1953, incorporate a further subsidiary company, Cannon Industries Ltd. This company was formed in order to provide a separate and more specialised direction and administration than was possible under the previous constitution. Since the completion of the financial year two further wholly owned subsidiary companies had been formed, Cannon (GA) Ltd.—Gas Appliance Sales and Service—and Cannon (CP) Ltd.—Chemical Plant Sales and Service. These companies took over from the parent company all matters of trade in their respective businesses, leaving the parent company the function of financial administration, investment, acquisition and general direction of the group's activity.

The chemical plant section of the business, now Cannon Industries Ltd., and Cannon (CP) Ltd., were during the year affected by deferred delivery and order placing. This was due to a cautious attitude created by a recession. The business finished the year with turnover and profit comparable with those of the previous year.

Sulphuric acid Directorate closed

With the revocation of the statutory controls on sulphuric acid and on sulphur used in its manufacture the Directorate of Sulphuric Acid and Sulphur Supplies has been closed. Enquiries on matters previously dealt with by the Directorate should be addressed to the Ministry of Materials, Branch 2A, Horse Guards Avenue, Whitehall, London, S.W.1.

Course on rarer base metals

A special course on The Rarer Base Metals, Their Production and Modern Industrial Applications is being held at Acton Technical College, London, W.3. The lectures, which cover most of the Rarer Base Metals, are given by experts engaged in extraction and production of these metals, and in addition the properties and present and potential uses of the metals in industry are dealt with. The lectures are of value to chemists, metal-

lurgists and others engaged in industry or research, and who are interested in modern extraction methods, and the many and varied applications of these metals.

The lectures began in January and the last lecture in the course will be given on March 26.

GERMANY

Plastics industry's expansion

Production, turnover and exports of the West German plastics industry have risen steadily since last spring. New orders booked during recent months have also been satisfactory. Since the beginning of 1953 exports have been 25% higher than in 1952 and are expected to reach a post-war peak for the whole year.

The industry experienced wide fluctuations in raw material supplies in 1953. Demand for some polymerisation productions, such as polystyrene and polyvinyl chloride, could not be met entirely and there was a severe shortage of polythenes.

Of the 67 West German plastics producers, 45 are medium-sized and small plants, which also manufacture other articles. Only nine firms in West Germany produce plastics exclusively. The 22 large producers account for 80% of the output of plastics and allied products. It is anticipated that the volume of production will increase over the next few years in favour of the large producers which manufacture their own raw materials.

Dechema Institute opened

The Dechema-Institut für Apparate- und Stoffkunde (Dechema Institute for Chemical Apparatus and Materials) was formally opened in Frankfurt-am-Main recently.

The Institute represents an attempt to illustrate in a practical manner the broad area covered by the field of chemical apparatus and equipment. The Institute will classify in its Register all chemical apparatus and equipment with respect to its theoretical bases of design, its construction, mode of operation and application to industry. A compilation of 100 materials with full particulars of their physical properties and resistance to 1,000 chemicals is now appearing in a Third Edition.

AUSTRIA

New paper plant

The new paper plant of the Murztaler Holzstoff und Papierfabrik A.G. at Bruck-an-der-Mur, Styria, has recently been completed. The plant has an output capacity of 120 tons of newsprint daily. Construction began in 1950 and was financed to a large extent by European Recovery Programme credits.

MEXICO

New sulphur plant in operation

Production of sulphur has started at the San Cristobal plant of the U.S.-owned Mexican Gulf Sulphur Co. in Vera Cruz State, Mexico. It is understood that the firm has brought in two additional wells, which are now being steamed.

SPAIN

New aluminium fabricating factory

Construction of a new fabricating factory for aluminium is well under way at Alicante. The new factory is owned by Aluminio Iberico, S.A., a company controlled jointly by Aluminium Ltd. of Canada, Manufacturas Metalicas Madrilenes, S.A., of Madrid, and a group of Spanish banks and individuals. It will make aluminium sheet plate and extrusions, aluminium steel-reinforced cables for power transmission and aluminium foil, and will have an output capacity of from 18,000 to 20,000 tons p.a. of these products. Ingots will be supplied by smelting subsidiaries of the Canadian firm.

SOUTHERN RHODESIA

Uranium mine planned

It is intended to float a company to establish Central Africa's first uranium mine near Beit Bridge. Uranium ore deposits there have been tested by the Atomic Energy Division of the British Geological Survey and it is reported that they have been taken over by a Johannesburg mining concern.

AUSTRALIA

'Catcracker' for Australia's largest refinery

Construction work on one of the first catalytic cracking units to be built in Australia is planned to begin soon at Geelong Refinery, near Melbourne. Scheduled for completion about the middle of 1955, the new plant will have an intake capacity of 650,000 tons p.a. and will convert heavy oil fractions to motor spirit, thus making an important contribution to Australia's requirements of this commodity.

This project forms part of a construction programme designed to make Geelong one of the largest and most modern oil refineries in the Commonwealth. The first stage in the programme, comprising a crude distillation unit with a capacity of approximately 1¾ million tons p.a. and ancillary treating facilities, is expected to be completed in the spring.

The catcracker complex will include a feed preparation unit to prepare the feedstock for the 'catcracker' proper and later a polymerisation plant for converting a portion of the valuable catcracked gases to high-grade motor spirit. When this complex is completed Geelong's tankage will consist of some 60 storage tanks with a capacity of approximately 400,000 tons.

In addition, the Geelong Harbour Trust is dredging the port approaches to a depth of 32 ft. and constructing a special jetty to accommodate ocean-going tankers of up to 18,000 deadweight tons.

Under present plans crude oil for the refinery will come from the Middle East and the Seria oilfield of British Borneo, both of which are virtually 100% sterling sources of supply.

Geelong Refinery is one item in a £100 million refinery construction programme which is being undertaken by the oil industry in Australia. Like all the operations of what is essentially an international industry, the technique and 'know-how' that has made Geelong possible is derived from many countries, but Australians are playing a major role in the building of the refinery and form a large proportion of the men at work on the site.

SWEDEN

Salt deposits investigated

Sweden may become self-supporting in salt through new finds in the province of Scania, according to a member of the Swedish Geological Research Institute. The Institute have applied to the Government for 500,000 crowns to complete research connected with the discoveries. So far geologists have penetrated to a depth of 1,600 metres at Hoellviken and found water with a 10% edible salt and 6% low quality, non-edible salt content.

Pulp project

The Forest Owners' Association of the South Swedish province of Scania has decided to build a 'semi-chemical' pulp factory at an estimated cost of 7 million crowns. A company has been formed for the purpose, the participants being the F.O.A., private forestry owners and certain other firms interested in the project.

The factory will have a capacity of 17,000 tons p.a. and will use about 90,000 cu. m. of timber and waste material from the sawmills.

FRANCE

Sulphur from natural gas

Sulphur is being produced from natural gas for the first time in France at the Berre-L'Etang (southern France) refinery of the French oil concern, Compagnie de Raffinage Shell-Berre. Production started on November 13 and will, it is expected, total between 4,000 and 5,000 tons during 1954.

Present production of sulphur in France totals about 15,000 tons p.a., against domestic requirements amounting to some 50,000 tons. The difference has been made up in the past by imports from the United States and Italy, so that the new domestic source of supply will mean a substantial saving in foreign currency.

New chemical enterprise

Two prominent French chemical firms, 'Pechiney' and 'Progil,' have founded a joint company, 'Pechiney-Progil (Société pour le Dévelopement et la Vente des Specialités Chimiques)' with a capital of 200 million francs, 100 million supplied by each of the companies. The new company's aim is to produce, buy, make up and sell chemical products. It will replace and extend the firm 'Sadex' which the two companies managed previously for the sale of chemicals for agricultural uses. The new company will act for its own account by manufacturing certain products with chemicals produced by the founders.

CANADA

Chlorine plant starts production

A $3½-million caustic chlorine plant built by Western Chemicals Ltd. has started production at Duvernay, on the North Saskatchewan River about 70 miles east of Edmonton. The plant manufactures chlorine, caustic soda and hydrochloric acid. Natural gas from the company's own wells will supply the plant with fuel, and natural salt will come from other company-owned property. Salt is the main raw material in manufacture of the chemicals.

Present daily capacity of the plant is 10 tons of chlorine, 12 tons of caustic soda and 10 tons of hydrochloric acid. This is to be expanded later.

Union Carbide changes

Union Carbide Canada Ltd., a subsidiary of Union Carbide and Carbon Corporation, is now operating the business of Bakelite Co. (Canada) Ltd., Canadian Railroad Service Co. Ltd., Carbide and Carbon Chemicals Ltd., Dominion Oxygen Co. Ltd., Electro Metallurgical Co. of Canada Ltd. and National Carbon Ltd.

These companies are units of Union Carbide and Carbon Corporation. In the future their respective businesses will be carried on by divisions of Union Carbide Canada under the following division names: Bakelite Co., Canadian Railroad Service Co., Carbide Chemicals Sales Co., Dominion Oxygen Co., Electro Metallurgical Co. and National Carbon Co.

Mr. Ewart Greig, formerly vice-president of National Carbon Ltd., is the president and director of Union Carbide Canada Ltd.

INDIA

Plant makers urged to unite

The formation of an association of chemical plant manufacturers in India, on similar lines to those of the B.C.P.M.A. in the U.K., is suggested by Mr. H. Pickard, managing director of the A.P.V. Engineering Co. Ltd., Calcutta, in a letter to *Chemical Age of India*.

One of its aims, he suggests, should be to provide training schemes 'to utilise the present surplus of graduate chemical engineers—many without any practical experience.'

Big steel plant planned

The Indian Government and the German combine of Krupps & Demag have signed agreements for setting up a 700 million rupee steel plant in India. The plant will have an initial capacity of half a million tons annually, rising by stages to a million tons. A company called Hindustan Steel Ltd. with an authorised capital of 1,000 million rupees will be floated to erect and run the plant. The new company will have a first capital of 500,000 rupees, 400,000 rupees being contributed by the Indian Government and the rest by the combine.

German experts have already visited four possible sites—in West Bengal, Bihar, Orissa and Madhyapradesh.

The Leonard Hill Technical Group—February

Manufacturing Chemist—The Complexones in Pharmaceutical Analysis; Cost Control in the Chemical Industry, 3; The Pyridine Drugs; Pressure Measurement in the Chemical Industry; Emulsifiers made in Situ; Progress Reports on Economic Poisons, and Disinfectants.

Paint Manufacture—Dehydrated Castor Oil; Synthetic Colours; Priming Plasters; History of Paints, 3; How Inks Dry, 2.

Food Manufacture—Barclay Perkins Brewery; The Milling Industry; American Flavours for Soft Drinks.

Fibres—Some Applications of Radioactive Isotopes in Wool Research; Mothproofing Agents and their Application; Improving the Dyeing Properties of Polyacrylonitrile Fibres; Asbestos—the Mineral Fibre.

World Crops—Crop Prospects in the South Pacific; The Fate of Collective Agriculture in Yugoslavia; Mediterranean Grassland and Fodder Research; The Tropics as Sources of Vegetable Oils and Fats.

Petroleum—Oil and Gas Storage Vessels; Fuel Oils: their Efficient and Smokeless Combustion; Cathodic Protection of Pipelines and Storage Tanks—A Survey; Petroleum Abstracts.

Building Materials Digest—Varieties and Applications of Lightweight Concrete; Fibre Building Board, 2; Short Survey of Reconstruction in France; Trials with Aluminium Curtain Walls in the U.S.A.

Muck Shifter—A Grand Contour Canal; A Giant Quarry Plant on Wheels.

Dairyman—A Vacuum Tank with Pipeline Milking; Cost Figures on Bulk Milk Handling; A Survey of World Dairying, 4; Early Cream Separators and Farmhouse Buttermaking; A New Milk Receiving Installation; Pasteurised Milk by the Cummings Process; Air Conditioning for Dairies, 2.

IRAQ

American company to seek sulphur

A partnership agreement for the exploitation of the sulphur resources of Iraq is expected to be signed soon between the Iraq Government and the Texas Gulf Sulphur Co. This brings two years of negotiation to an end. According to the London *Times*, the agreement will give the company the right to seek and exploit the sulphur resources in all Iraq territory north of the 33rd parallel for 50 years. The profit will be shared on a fifty-fifty basis. The country's sulphur resources are believed to be considerable, and the agreement means setting up a new industry with a big dollar investment.

The company will pay the Iraq share of the profits in dollars and will provide sulphur for local use at a reduced rate. It is bound to hand back one-quarter of the total area every five years and this will be at the free disposal of the Government. At the end of 20 years the company will retain only 1,000 sq. km. It is believed that the agreement will open a new source of sulphur to Britain, which now buys from the United States.

VENEZUELA

Cement factory contract for Italy

The Genoa concern 'Ansaldo' has signed a contract with Venezuela for the construction of the machinery and electric plant for a cement factory. Total cost will be about 1,000 million lire. The Venezuelan factory will have a daily capacity of 450 tons of cement.

NETHERLANDS

New smokeless fuel

The Dutch State mines expect to be able to market soon a new smokeless and odourless fuel called *Synthracite*. It is claimed to equal best quality anthracite. For the time being it will be produced only on a small scale, but later an expansion of production capacity is planned. It is predicted that this 'synthetic anthracite' will prove to be of great importance as an addition to Holland's domestic fuel supplies. At present Dutch mines are unable to meet the great demand for anthracite. Production of *Synthracite* may eventually serve to ease this shortage.

UNITED STATES

Paper caps for chemical workers

A disposable working cap, made of heavy kraft paper which has been treated with neoprene synthetic rubber, has been developed by E. I. du Pont de Nemours & Co. for use in one of the company's large chemical plants. Savings through use of the paper caps are expected to run upwards of $2,000 p.a. Because of the possibility of dripping acids and other chemicals, about 3,000 workers at the plant are required to wear some sort of headgear. Formerly 15,000 cloth caps, intended for laundering and re-use, were issued annually by the company. However, laundering proved unsatisfactory and most of the caps were discarded after first use.

The new cap costs about a third less than the customary cloth caps, is water-repellent and is pretreated with a flame retardant. A small percentage of neoprene latex added to the pulp during the paper-making process is said to give the finished paper unusually high wet strength.

Organic peroxides from pine gum

A simple, rapid and economic process for producing pinane hydroperoxide from gum turpentine in good yield and good concentration, has been developed by scientists associated with the Naval Stores Station of the U.S. Department of Agriculture at Olustee, Florida. The new process will make pine gum—a plentiful crop—a potential source of organic peroxides. Pinane hydroperoxide is of particular value as a polymerisation catalyst in the production of synthetic rubber, resins and plastics.

Several chemical companies have shown interest in producing pinane hydroperoxide and other turpentine peroxides. These could, in an emergency, replace cumene hydroperoxide, a benzene compound, used as a catalyst in cold rubber production.

MEETINGS

Institution of Chemical Engineers

February 13. 'The Mixing of Solid Particles and Gas in a Fluid Bed,' by G. H. Reman, 2.30 p.m., Radiant House, Bold Street, Liverpool.

March 2. 'Design Aspects of Vinyl Chloride Manufacture,' by A. H. Goodliffe and Ir Noorduyn, 5.30 p.m., Geological Society, Burlington House, London, W.1.

Society of Chemical Industry

Chemical Engineering Group

February 9. 'Non-Ferrous Metals for the Chemical Engineer,' by N. P. Inglis, 5.30 p.m., Geological Society, Burlington House, London, W.1.

Incorporated Plant Engineers

February 11. 'Metallurgy,' by J. Rolston, 7 p.m., Roadway House, Oxford Street, Newcastle-on-Tyne.

February 15. 'Mechanical Service Equipment in a Medium Sized Chemical Factory,' by D. W. Evans, 7.15 p.m., Radiant House, Bold Street, Liverpool.

February 17. 'The Generation of Industrial Power from Process Steam,' by E. G. Ritchie, 7.15 p.m., Grand Hotel, Bristol.

Institute of Metals

February 9. 'The Zinc Die-Casting Alloys,' by L. A. J. Lodder, 6.45 p.m., University College, Metallurgy Department, Singleton Park, Swansea.

February 11. 'Diffusion in Metals,' by A. D. Le Claire, 7 p.m., Liverpool Engineering Society Rooms, The Temple, Dale Street, Liverpool.

March 2. 'Thermochemistry of Alloys,' by A. R. Harding, 7 p.m., Oak Room, Cadena Cafe, Cornmarket Street, Oxford.

March 2. 'Alumino-Thermic Methods,' by T. Burchell, 6.45 p.m., University College, Metallurgy Department, Singleton Park, Swansea.

March 3. 'Surface Finishing by Liquid Honing,' by D. V. Grant, 6.30 p.m., Lecture Room, The Central Library, Manchester.

March 4. 'High Temperature Corrosion,' by J. P. Dennison, 6.30 p.m., 4 Grosvenor Gardens, London, S.W.1.

March 4. 'Application of Aluminium and its Alloys,' by J. C. Bailey, 7.15 p.m., Chemistry Department, The University, Leeds, 2.

The Chemical Society

February 12. 'Photochemical and Spectroscopic Methods of Studying Fast Reactions,' by Pro. R. G. W. Norrish, 5 p.m., Chemistry Department, The University, Southampton. Joint meeting with the R.I.C.

February 15. 'Corrosion Theory in Practice,' by Dr. T. P. Hoar, 6.30 p.m., Chemistry Lecture Theatre, The University, Leeds.

February 15. 'Extraction of Chemicals from Sea Water,' by Dr. R. O. Gibson, 5 p.m., University College, Leicester.

February 17. 'Krilium Soil Conditioner,' by Dr. A. J. Lloyd, 7.15 p.m., Agriculture Lecture Theatre, Queen's University, Belfast. Joint meeting with the S.C.I.

February 18. 'Recent Progress in the Study of Metallic Corrosion,' by Dr. W. H. J. Vernon, 8 p.m., North British Station Hotel, Edinburgh. Joint meeting with the R.I.C. and the S.C.I.

February 19. 'Usnic Acid and Related Topics,' by Prof. A. Robertson, 8.30 p.m., Chemical Laboratory, The University, Cambridge.

February 25. 'Steel,' by Sir Charles Goodeve, 5.15 p.m., Chemistry Department, University College, Dundee.

February 26. 'Adhesion and Surface Properties of Solids,' by Dr. F. P. Bowden, 5.30 p.m., Chemistry Building, King's College, Newcastle-on-Tyne.

March 4. 'National Fuel Resources and their Utilisation,' by Dr. A. Parker, 7 p.m., Department of Chemistry, The University, Bristol. Joint meeting with the R.I.C., S.C.I., and the Institute of Fuel.

March 5. 'Rubber,' by Dr. C. M. Blow, 5.30 p.m., Technical College, Plymouth. Joint meeting with the R.I.C., and the S.C.I.

March 5. 'Chemical Engineering and Industrial Productivity,' by Prof. D. M. Newitt, 7.15 p.m., Agriculture Lecture Theatre, Queen's University, Belfast. Joint meeting with the R.I.C.

Royal Institute of Chemistry

February 10. 'The Production and Properties of Zirconium and Titanium,' by G. L. Miller, 7 p.m., South-West Essex Technical College, Forest Road, London, E.17.

February 17. 'Problems in the Industrial Application of Nuclear Energy,' by Sir W. A. Akers, 6 p.m., University College Mechanical Engineering Lecture Room, Gower Street, London, W.C.1.

February 24. 'The Production of Sulphuric Acid from Calcium Sulphate,' by W. L. Bedwell, 6.30 p.m., Norwood Technical College, Knight's Hill, S.E.27.

February 25. 'Some Uses of Radioactive Materials in Industry and Medicine,' by L. C. Myerscough, 6.30 p.m., South-East Essex Technical College, Longbridge Road, Dagenham, Essex.

March 3. 'A Century of Ion Exchange,' by E. J. Akeroyd, 6.30 p.m., West Ham Municipal College, London, E.15.

Institute of Fuel

February 17. 'Underground Gasification,' by C. A. Masterman, 7.30 p.m., Grosvenor Hotel, Chester.

February 23. 'Carbonisation of Blends of Coals to Produce Metallurgical Coke,' by H. Bardgett, 5.30 p.m., Institution of Mechanical Engineers, Storey's Gate, St. James's Park, London, S.W.1.

Institute of Petroleum

February 22. 'Oil Production in the Nottinghamshire Oilfields,' by C. M. Adcock and R. K. Dickie, 5.30 p.m., 26 Portland Place, London, W.1.

Institution of Mechanical Engineers

March 3. 'Pressure Surge in Pump Pipe Lines,' 6.45 p.m., Storey's Gate, St. James's Park, London, S.W.1.

Institution of Rubber Industry

February 8. 'Silicone Rubbers,' by R. B. Noad, 6.45 p.m., James Watt Institute, Gt. Charles Street, Birmingham.

February 8. 'Rubber-based Adhesives,' by A. A. Round, 6.45 p.m., James Watt Institute, Gt. Charles Street, Birmingham.

February 22. 'Variability of Natural Rubber in the Presence of Carbon Black,' by H. C. Baker, 6.15 p.m., Engineer's Club, Albert Square, Manchester.

CHEMICAL & PROCESS ENGINEERING

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